THE LABORATORY BOOK

OF

MINERAL OIL TESTING

Griffin's Standard Publications.

THIRD EDITION, Revised Throughout. Reset on Larger Page. With Valuable Bibliography, New Maps, Illustrations, &c.

PETROLEUM AND ITS PRODUCTS.

A Practical Treatise.

By Sir BOVERTON REDWOOD, F.R.S.E., F.I.C., Assoc.R.C.S.
Hon. Corr. Mem. of the Imperial Russian Technical Society; Mem. of the American Chemical Society; Adviser to the Home Office and to the Corporation of London under the Petroleum Acts, &c. &c.

SECOND EDITION. With Plates (one Coloured) and Illustrations. 8s 6d net

A HANDBOOK ON PETROLEUM.

For Inspectors under the Petroleum Acts.

By Captain J. H. THOMSON,
H.M. Chief Inspector of Explosives,
And **Sir BOVERTON REDWOOD.**

"Reliable, indispensable, a brilliant contribution."—*Petroleum.*

SECOND EDITION. In Large Crown 8vo, Cloth. Fully Illustrated. 6s net

OIL FUEL:

Its Supply, Composition, and Application.

By SIDNEY H. NORTH.

Revised by EDWARD BUTLER, M.I.Mech.E.

"Every one interested in this important question will welcome Mr. North's excellent text-book."—*Nature.*

In Cloth Boards. Fully Illustrated. 1s net

THE PETROLEUM LAMP:

Its Choice and Use.

By Captain J. H. THOMSON

And **Sir BOVERTON REDWOOD.**

"A work which will meet every purpose for which it has been written."
Petroleum.

SECOND EDITION. In Large 8vo. Fully Illustrated. 25s net

OILS, FATS, BUTTERS, AND WAXES:

Their Preparation and Properties, and the Manufacture therefrom of Candles, Soaps, and other Products.

By C. ALDER WRIGHT, D.Sc., F.R.S.

Thoroughly Revised, Enlarged, and in Part Rewritten

By C. AINSWORTH MITCHELL, M.A., F.I.C.

"Will be found absolutely indispensable."—*Analyst.*

THIRD EDITION. Large 8vo. Cloth. Fully Illustrated. 25s net

LUBRICATION AND LUBRICANTS.

A Treatise on the Theory and Practice of Lubrication and on the Nature, Properties, and Testing of Lubricants.

By LEONARD ARCHBUTT, F.I.C., F.C.S.

And **R. MOUNTFORT DEELEY, M.I.Mech.E., F.G.S.**

"Destined to become a classic on the subject."—*Industries and Iron.*

In Large 8vo. Handsome Cloth. 16s net

FIRE AND EXPLOSION RISKS.

A Handbook on the Detection, Investigation, and Prevention of Fires and Explosions.

By Dr. VON SCHWARTZ.

"A wealth of information on the chemistry of fire and kindred topics."
Fire and Water.

LONDON: CHARLES GRIFFIN & Co. LTD., EXETER STREET, STRAND.

Abel Petroleum Testing Apparatus.

THE LABORATORY BOOK

OF

MINERAL OIL TESTING

BY

JAS. A. HICKS
LATE CHIEF CHEMIST TO SIR BOVERTON REDWOOD

WITH INTRODUCTION BY

SIR BOVERTON REDWOOD

(*THIRTY-TWO ILLUSTRATIONS*)

Second Edition, Revised

LONDON
CHARLES GRIFFIN AND CO. LIMITED
EXETER STREET, STRAND
1912

BALLANTYNE & COMPANY LTD
TAVISTOCK STREET COVENT GARDEN
LONDON

PREFACE TO SECOND EDITION

THE welcome accorded the first edition of this little book was most gratifying to the author, and the call for a second edition at so early a date found little need for revision. The author was able, however, to examine and carefully correct the text where necessary, also to add further notes before the sudden illness seized him from which, unhappily, he did not recover. By the courtesy of Sir Boverton Redwood, a note upon the latest design of his Viscometer is added.

C. G. & Co. LTD.

April 1912.

PREFACE TO FIRST EDITION

I HAVE been requested to compile the following notes on the commercial examination of mineral oil products as an outcome of a personal experience of some sixteen years' continual work on the subject.

I have not endeavoured to write a text-book, but to give in a concise form such details for the working of apparatus special to the testing of petroleum and its derivatives as will enable the analyst who may have occasionally to examine such bodies, to proceed in a satisfactory manner. At the same time it has seemed well to include those ordinary physical tests which require special modifications for this work.

JAS. A. HICKS.

May 1906.

INTRODUCTION

By Sir BOVERTON REDWOOD
D.SC. (HON.), F.R.S.E.
Adviser on Petroleum to the Admiralty and the Home Office.

The great importance of standardising the methods adopted in the examination of samples of commercial products and in the reporting of the results is unquestionable, for a large proportion of disputes between sellers and buyers are directly traceable to misunderstandings arising from differences of procedure.

To no branch of commerce is this remark more applicable than to the mineral oil industry, and no better illustration of it can be furnished than a reference to the chaotic character of the statements commonly made in respect of the viscosities of specified oils in the early days of that industry.

Within recent years much has been done to place the testing of mineral oil products upon a satisfactory basis by the adoption of uniform methods, but there is still, among some of those to whom this class of work is only occasionally entrusted, a lack of knowledge of certain of the instruments and processes which others,

whose avocation brings them wider experience of the requirements, have devised or selected as specially suited to the purposes.

In these circumstances the publication of a manual designed to convey the requisite information needs no justification, and I do not know any one better qualified to undertake the authorship of such a work than Mr. J. A. Hicks, who has had daily experience of the various operations in question for the past sixteen years.

Mr. Hicks has written an unassuming book which cannot fail to prove a most valuable practical guide to analytical chemists and others who are called upon to perform the class of work dealt with.

CONTENTS

PRELIMINARY.

THE commercial examination of Mineral Oil Products may be said to divide itself roughly into that which is made in connection with legal requirements as to storage and so forth, and that which aims at satisfying the purchaser as to the quality of the sample for practical use.

The principal tests employed are those for specific gravity, for flashing-point, and, in the case of lubricating oils, for viscosity. But further trials are made of some commercial products manufactured for special purposes, which may or may not require apparatus additional to that usually found in the laboratory of the general chemist.

The following table of the results commonly yielded by the different products will be of use as a guide to what may be looked for :—

Product.	Sp. Gr. at 60° F.	F.P. (close) °F.	Viscosity (Redwood).		B.P. °F.	
			Secs.	R.O. at 60° F. =100.		
Pentane . .	·624–·626	Below			77–99	
·650 Gasoline .	·642–·648	0° F.			90–200	
Petroleum Ether . .	·630–·730	,,			80–300	
·680 Spirit. .	·660–·700	,,			120–250	
Benzoline (Benzine) .	·690–·720	,,			130–350	
Ligroine . .	·715	,,			190–250	
Petrol . .	·715–·750	,,			130–300	
Kerosene :						*Colour.*
American :						(Wilson).
Ordinary .	·798–·802	73–80				2·6–3·0
High Test .	·785–·792	100–110				1·7–2·0

Product.	Sp. Gr. at 60° F.	F.P. (close) °F.	Viscosity (Redwood). Secs.	R.O. at 60°. F. = 100.	B.P. °F.	
Kerosene :						*Colour.* (Wilson.)
Russian:						
Ordinary .	·822–·827	85–88				2·1–2·4
Mineral Sperm	·825	250				
Pyronaphtha .	·865	250				
Gas Oils :						
American . .	·885–·865 (and beyond).	100–150				
Russian (Solar oil) . .	·870–·880	210–240				
Lubricating Oils :						
Spindle :			at 70° F.			
American	varies.	350–370	180	32		
Russian .	·897–·898	340–350	330–360	60–66		
Engine :						
American	varies.	360–400	500–750	90–130		
Russian .	·908–·909	380–400	1150–1300	210–240		
Cylinder :			at 200° F.			
American	varies.		varies.			
Russian .	·914–·915	430–440	75	13–14		
Astatki :			at 70° F.			
Russian .	·911–·913	310–340	1700–2300	310–430		
Paraffin :						M.P. (*English*).
Scale . .						110–125°F.
Wax . .						125–130°F.

CHAPTER I.

SPECIFIC GRAVITY.

THE determination of the specific gravity of petroleum products requires special care in recording the temperature of working, owing to the high coefficient of expansion possessed by these bodies. It is usually taken at 60° F. (15·56° C.), or is corrected to that standard by the following coefficients.

Spirits lighter than Kerosene	·00048 to ·00040 for 1° F. dependent on the volatility of the sample.
Kerosene,	·00040.
Gas Oils,	·00036.
Lubricating Oils,	·00034.

These figures include the correction for the expansion of the glass vessel used in the determination, and are correct when used to adjust to the standard from the temperature of determination; but to correct the specific gravity of the oil itself from one temperature to another a coefficient 0·00001 higher must in all cases be taken.

Hydrometer.—For thin oils the hydrometer is commonly considered accurate enough for most purposes if it has a sufficiently open scale. A form such as that shown in Fig. 1 is an improvement on that commonly employed. The bulk of the liquid necessitated by its use (12 ounces) makes it advisable to stir well with the thermometer before and after the reading is taken. Such a hydrometer is conveniently made with a range of 0·02, and this being distributed over a scale six

inches in length makes readings possible to the fourth place of decimals with fair accuracy. When first employed it should be standardised with oils whose specific gravity has been exactly determined in the specific gravity bottle ; and these oils should be of such densities as to afford readings generally distributed over the whole range of the scale, for the correctness of the instrument commonly varies with different densities. The hydrometers may conveniently read from ·780 to ·800, from ·800 to ·820, from ·820 to ·840, from ·840 to ·860, and from ·860 to ·880, respectively.

Fig. 1.—Hydrometer.

Baumé and Twaddell Hydrometers.—The scales of most hydrometers in use in this country are marked in terms of what is known as "absolute specific gravity," that is, they show readings that compare the weight of a unit volume of the sample with that of a unit volume of water. Unfortunately, other scales are also in existence, notably those of Baumé and Twaddell, which are quite arbitrary in their divisions.

To convert **Twaddell** degrees to specific gravity, multiply by 5 and deduct the product from 1000. To find the specific gravity equivalent of **Baumé** degrees, add 130 and divide 140 by the sum. The first of the following tables gives the absolute specific gravity equivalents of the Baumé degrees required for oils and spirits, and was obtained by the above formula.

The second table, which is in use in the United States, will be seen to differ considerably from the first, but it must be borne in mind that in some makes of the Baumé hydrometer the factor here given as 130 is as high as 135.

TABLE I.

Baumé.	Sp. Gr.	Baumé.	Sp. Gr.	Baumé.	Sp. Gr.
10	1·0000	41	·8187	72	·6931
11	·9929	42	·8139	73	·6897
12	·9859	43	·8092	74	·6863
13	·9790	44	·8046	75	·6829
14	·9722	45	·8000	76	·6796
15	·9655	46	·7955	77	·6763
16	·9589	47	·7910	78	·6731
17	·9524	48	·7865	79	·6699
18	·9460	49	·7821	80	·6667
19	·9396	50	·7777	81	·6635
20	·9333	51	·7734	82	·6604
21	·9272	52	·7692	83	·6573
22	·9211	53	·7650	84	·6542
23	·9151	54	·7609	85	·6512
24	·9091	55	·7568	86	·6482
25	·9032	56	·7527	87	·6452
26	·8974	57	·7487	88	·6422
27	·8917	58	·7447	89	·6392
28	·8861	59	·7407	90	·6363
29	·8805	60	·7368		
30	·8750	61	·7330		
31	·8696	62	·7292		
32	·8642	63	·7254		
33	·8589	64	·7217		
34	·8537	65	·7180		
35	·8485	66	·7143		
36	·8434	67	·7107		
37	·8383	68	·7071		
38	·8333	69	·7035		
39	·8284	70	·7000		
40	·8235	71	·6965		

TABLE II.

Baumé.	Sp. Gr.	Baumé	Sp. Gr.	Baumé.	Sp. Gr	Baumé.	Sp. Gr.
10	1·0000	30	·8755	50	·7794	70	·7025
11	·9930	31	·8702	51	·7752	71	·6990
12	·9861	32	·8650	52	·7711	72	·6956
13	·9791	33	·8597	53	·7670	73	·6923
14	·9722	34	·8544	54	·7628	74	·6887
15	·9658	35	·8492	55	·7587	75	·6856
16	·9594	36	·8443	56	·7546	76	·6823
17	·9530	37	·8395	57	·7508	77	·6789
18	·9466	38	·8346	58	·7470	78	·6756
19	·9402	39	·8299	59	·7432	79	·6722
20	·9339	40	·8251	60	·7394	80	·6689
21	·9280	41	·8204	61	·7357	81	·6656
22	·9222	42	·8157	62	·7319	82	·6619
23	·9163	43	·8110	63	·7281	83	·6583
24	·9105	44	·8063	64	·7243	84	·6547
25	·9047	45	·8017	65	·7205	85	·6511
26	·8989	46	·7971	66	·7168	86	·6481
27	·8930	47	·7927	67	·7133	87	·6451
28	·8872	48	·7883	68	·7097	88	·6422
29	·8814	49	·7838	69	·7061	89	·6363

Specific Gravity Bottle.—The ordinary Specific Gravity Bottle with a drilled stopper, having a capacity of 50 c.c., is the one generally employed where greater accuracy is required than is possible with a hydrometer. But, of course, where the sample is of less volume than 50 c.c., bottles of 10 c.c. or 25 c.c. can be adopted, if a less accurate result is sufficient.

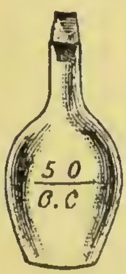


FIG. 2.—Specific Gravity Bottle.

A narrow-stemmed thermometer, marked in half-degrees F., is necessary, and temperatures should be read to a quarter of a degree F.

The bottle is filled with the sample in question, and the thermometer inserted. When the air-bubbles have totally dispersed, and the temperature has become constant (the latter must be ascertained by stirring with the thermometer from time to time), the thermometer is

removed, the space left by it being filled up with a few drops of oil from a small bulk which has been standing in close proximity to the bottle.

The stopper is then put in and pushed well home with a slight rotary motion, the excess of liquid which flows through the drilled hole is carefully wiped away with a piece of filter-paper, and the surface of the oil is left exactly on a level with the top of the stopper. From the time the thermometer is removed to this point the bottle must not be handled in the slightest, and the hands should not approach it to a greater extent than is necessary, only the tips of the fingers being used for operating. The bottle is now carefully wiped with a soft cloth and weighed at once on a delicate balance. The tare of the bottle and stopper, which has been ascertained previously, is deducted from the weight found, and the remainder is divided by the water-contents of the bottle at 60° F., which has been also very exactly determined beforehand. The quotient is the specific gravity of the sample at the temperature of working. The tare and water-contents of the apparatus should be checked from time to time, distilled water, of course, being employed.

In the case of **Spirits** it is essential that great care be taken that none of the liquid is lost by the partial evaporation of the portion which may expand out of the bottle during wiping. This is best avoided by adjusting in a beaker of water slightly warmer than the atmosphere of the laboratory, so that after the top of the stopper is wiped and the bottle is removed from the bath the spirit contracts slightly down the drilled hole, and the bottle can be manipulated safely. This use of a water-bath also obviates the awkward variations of temperature caused by the evaporation of spirit which may have got on to the outside of the bottle.

With **Lubricating Oils** of moderate viscosity it is sufficient to follow the above directions, but when working with those of a thicker nature the sample must be thoroughly warmed before it can be poured

into the bottle, which, when full, should be placed in a steam oven for some time to ensure the entire absence of air. When this is certain, it is taken out, allowed to cool slowly and finally placed for two or three hours in a beaker of water that also holds the thermometer. If this be not done and the thermometer be put in the bottle in the usual way, it is almost impossible to avoid the air, which passes in when it is withdrawn, being trapped by the oil and vitiating the result.

Some **Cylinder Oils** present a peculiar difficulty when cooling, owing to the sample solidifying in the neck before contraction has ceased in the body of the bottle, and an air passage is forced through the solid, leading to a bubble in the interior. This can be most satisfactorily avoided by cooling slowly, and, on any signs of solidification becoming apparent, by stirring the oil in the neck with a warm wire until contraction is complete. With such samples very great care must be taken when placing the stopper in position, or the bottom of the bottle will be forced out.

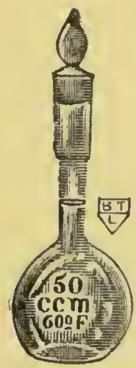


Fig. 3.—Regnault Specific Gravity Bottle.

Regnault Bottle.—When the common specific gravity bottle contains **very thick liquids** and the drilled stopper is inserted, danger of bursting is incurred. With the Regnault Bottle this is avoided, as the contents are adjusted to a mark on the neck.

The best method of working, perhaps, with very thick oils is to fill the bottle with a warmed portion of the sample, and when cold to remove some of the contents and thoroughly clean the neck to a point a millimetre or so below the mark. Placing the bottle then in a beaker of water and very gradually warming will bring the meniscus up to the line, and a thermometer in the water will give the required temperature. Very viscous oils frequently leave a hollow core of air down the neck in cooling, and as

this often means the formation of an invisible air-bubble, the oil in the neck must be kept warm until the bottle has reached a stationary temperature. This may be done by stirring with a *warm* wire. Removal of the surplus oil can be conveniently accomplished with a strip of stiff filter-paper, folded down the middle and cut diagonally at one end, and the cleaning, with a roll of the same.

The Regnault Bottle being entirely closed by its stopper makes it very convenient for use with **spirit**, as after the stopper is inserted no evaporation can take place. In such use the liquid may, of course, be adjusted to the contents mark directly, and without cooling and rewarming as with cylinder oils.

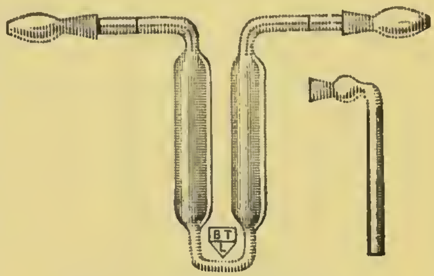


FIG. 4.—Sprengel Tube.

Sprengel Tube.—An ingenious modification of the bottle consists of a U-tube with horizontal extremities of capillary tubing. It is particularly useful for the determination of **specific gravities at elevated temperatures,** as the peculiar form enables it to be suspended in a bath, leaving the ends projecting for adjustment.

To fill, remove the closing caps, replace one with the filling tube, and connect a few inches of rubber tubing to the other. Invert the U-tube, letting the filling tube project downwards into the sample, which, if very viscous, may have been warmed. Then exhausting the air by suction of the rubber will cause the first limb of the U-tube to be filled. As soon as this is complete turn the tube right side up, without removing the filling tube from the sample, but revolving it upon its ground-glass connection. Proceed with the exhaustion until the oil has completely filled the apparatus. Turn the filling tube up so that it stands uppermost, immerse the U-tube in a bath with a thermometer, and raise to the

required temperature. Leave the whole until the temperature is uniform, then remove the filling and rubber tubes, also the surplus oil from the ends of the capillaries, replace the caps and weigh after wiping. A platinum wire hook will be found useful as a support on the balance.

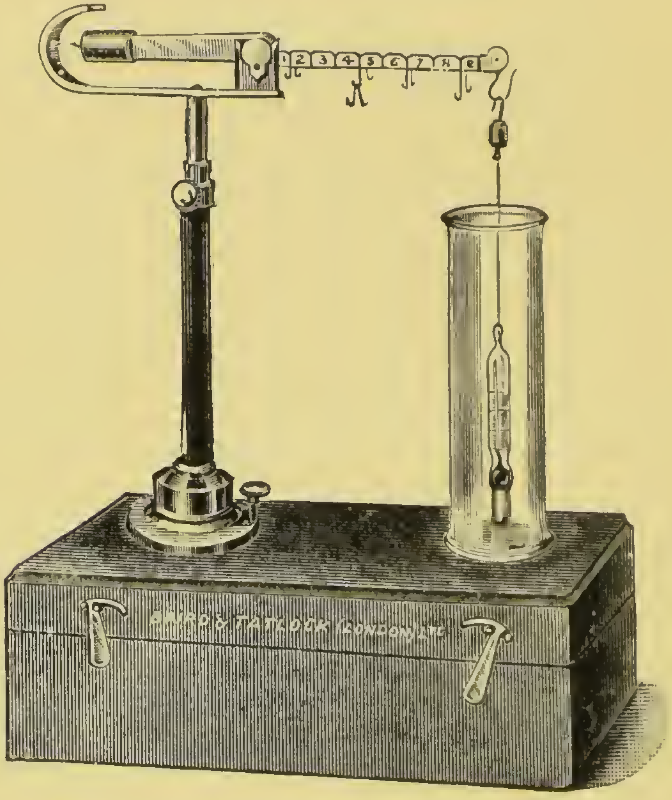


FIG. 5.—Westphal's Specific Gravity Balance.

Westphal Balance.—The Westphal Balance is of use for oils the viscosity of which is not sufficiently high to interfere with the free movement of the plummet or float. Stirring with an accurate thermometer before and after each reading and a careful observation of the size of the different riders and their respective positions on the beam, will give figures trustworthy to about two or three in the fourth place of decimals. It is best to disregard the thermometer contained in the plummet, as

the scale is not sufficiently open. The largest sized rider represents ·1 when hung on the notch marked "1"; when on notch "2," ·2, and so on. The other riders represent ·01, ·001, and ·0001 according to their size, and all are usually supplied in duplicate.

To adjust the balance, the foot is turned round until the levelling screw is under the plummet end of the beam, and with the plummet hanging in air the screw

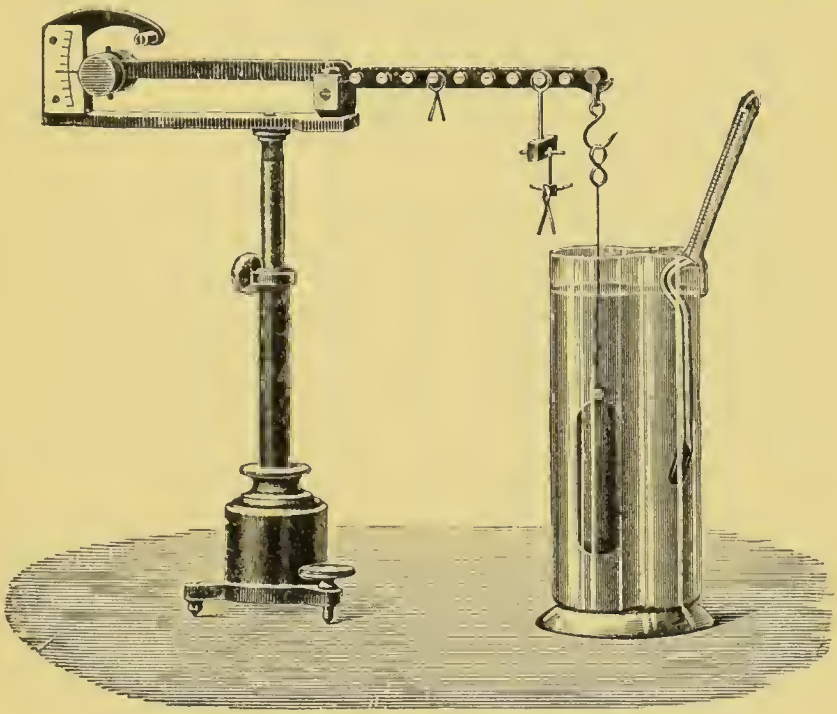


FIG. 6.—The Sartorius Specific Gravity Balance.

is turned up or down until the needle-point on the counterpoise bob vibrates to an equal extent on either side of the corresponding point on the frame.

Sartorius Balance.—The Westphal Balance has been modified by several designers. It will be necessary here to notice only that of Sartorius.

The general notes as to the use of Westphal's Balance apply, with the addition that in the examination of oils it will be, perhaps, advisable to discard the double cylinder and to substitute one of the

usual shape. The readings obtained are more definite with this instrument than with the less expensive form, owing partly to the use of a larger plummet. A thermometer divided into half-degrees F. should be employed and read to quarter-degrees.

Very Small Samples.—The specific gravity of very small samples can be ascertained with a fair approach to accuracy by mixing alcohol and water to such a density that a drop of the oil placed in the mixture will show no tendency to sink or rise, and by then taking the specific gravity of the mixture by one of the above methods.

The addition of alcohol to water generates heat, and time must accordingly be given for the temperature of the mixture to approach that of the room after each addition of either liquid. The temperature at which the position of the drop is stable must be read, and the density of the mixture ascertained at that temperature.

CHAPTER II.

FLASHING-POINT.*

THE **flashing-point**, or **flash-point**, of an oil is the empirical temperature at which it gives off sufficient vapour to ignite momentarily on the introduction of a flame or spark, when the oil is heated at a given rate, in an apparatus of given construction and dimensions, and a defined igniting agent is applied in a given manner.

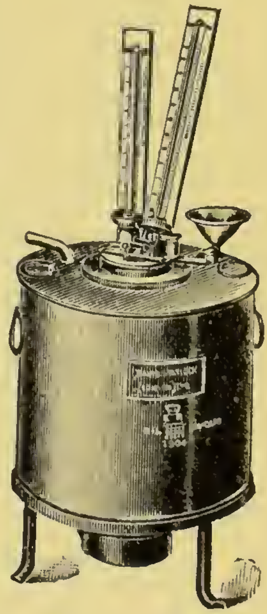


FIG. 7.—Abel Petroleum Tester.

The "flashing-point" of an oil will therefore vary with the different instruments used in its determination. The results yielded by these instruments may be divided into two classes, "close" and "open," the former being given by the forms of apparatus having a lid to the testing-cup, and the latter by those which have none. To these two classes must be added the "fire test," which differs from them by showing the temperature at which the vapour is evolved so rapidly as to continue burning when ignited.

Abel Petroleum Tester.—The legally recognised method of determining the flashing-point of an oil in

* Redwood, "Petroleum," ed. 1906, p. 545. Thomson and Redwood, "Handbook on Petroleum," ch. v. and vi.

the United Kingdom is that designed by Sir Frederick Abel and embodied in the Petroleum Act of 1879. The manipulation of the Abel Tester is there set forth as follows :

PETROLEUM ACT, 1879.

(42 & 43 Vict. c. 47.)

Mode of Testing Petroleum so as to ascertain the Temperature at which it will give off Inflammable Vapour.

The test apparatus should be placed for use in a position where it is not exposed to currents of air or draughts.

The heating vessel or water-bath is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be 130° F., and this is attained in the first instance either by mixing hot and cold water in the bath, or in a vessel from which the bath is filled, until the thermometer which is provided for testing the temperature of the water gives the proper indication ; or by heating the water with the spirit lamp (which is attached to the stand of the apparatus) until the required temperature is indicated.

If the water has been heated too highly, it is easily reduced to 130° by pouring in cold water little by little (to replace a portion of the warm water) until the thermometer gives the proper reading.

When a test has been completed this water-bath is again raised to 130° by placing the lamp underneath, and the result is readily obtained while the petroleum cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus, and the next is proceeded with.

The test lamp is prepared for use by fitting it with a piece of flat-plaited candle-wick,* and filling it with colza or rape oil up to the lower edge of the opening of the spout or wick table.

The lamp is trimmed so that when lighted it gives a flame of about 0·15 of an inch in diameter; and this size of flame, which is represented by the projecting white lead on the cover of the oil-cup, is readily maintained by simple manipulation from time to time with a small wire trimmer.

When gas is available it may be conveniently used in place of the little oil lamp, and for this purpose a test-flame arrangement for use with gas has been devised, which may be substituted for the lamp.

The bath having been raised to the proper temperature, the oil to be tested is introduced into the petroleum cup, being poured in slowly until the level of the liquid just reaches the point of the gauge which is fixed in the cup.† In warm weather the temperature of the room in which the samples to be tested have been kept should be observed in the first instance, and if it exceeds 65°, the samples to be tested should be cooled down (to about 60°) by immersing the bottles containing them in cold water, or by any other convenient method.

The lid of the cup, with the slide closed, is then put on, and the cup is placed into the bath or heating vessel. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position is not under any circumstances to be altered.

When the cup has been placed in a proper position, the scale of the thermometer faces the operator.

* The description of wick known as Field's night-light candle-wick has been found most suitable.

† Great care must be taken to prevent the oil being splashed against the sides of the cup and the formation of air-bubbles.

The test lamp is then placed in position upon the lid of the cup, the lead line or pendulum,* which has been fixed in a convenient position in front of the operator, is set in motion, and the rise of the thermometer in the petroleum cup is watched.

When the temperature has reached about 66° the operation of testing is to be commenced, the test flame being applied once for every rise of one degree in the following manner :

The slide is slowly drawn open while the pendulum performs *three* oscillations and is closed during the *fourth* oscillation.

NOTE.—If it is desired to employ the test apparatus to determine the flashing-points of oils of *very* low volatility, the mode of proceeding is to be modified as follows :

The air chamber which surrounds the cup is filled with *cold* water to a depth of 1½ inch, and the heating vessel or water-bath is filled as usual, but also with cold water.† The lamp is then placed under the apparatus and kept there during the entire operation. If a heavy oil is being dealt with, the operation may be commenced with water previously heated to 120°, instead of with cold water.

In the Petroleum Bill of 1883 the following remarks occur on the use of the pendulum :

The first oscillation is from *a* to *b*.
,, second ,, ,, ,, *b* to *a*.
,, third ,, ,, ,, *a* to *b*.
,, fourth ,, ,, ,, *b* to *a*.

The opening of the slide commences the moment the pendulum leaves position *a* in the first oscilla-

* Pendulum twenty-four inches long.

† A far preferable method, although not in accordance with the terms of the Act, is to pour water into the air chamber to a depth of ¼ inch, and to maintain the outer bath at 130° as usual.

tion and is steadily continued while it performs the first, second, and third oscillations, so that the slide is fully open when, in the third oscillation, the pendulum has reached position *b*. The slide is kept open for an instant and then quickly shut, the moment of its being quite closed again being coincident with the return of the pendulum to position *a* at the end of the fourth oscillation.

A clock having a 24-inch pendulum is useful for those who are continuously making the test.

In every case in which the test is repeated, a fresh portion of the sample must be used.

Although in English law there is no allowance for the barometric pressure prevailing at the time the test is made, it must be borne in mind that a difference in the height of the barometric column does effect a considerable alteration in the result of the test.

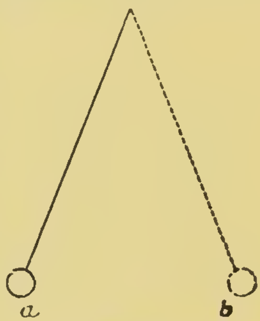


FIG. 7*a*.

The alteration thus brought about is shown in the table on page 18, which gives results as determined by the Abel instrument.

The use of the Abel Tester with liquids containing **solid matter in solution or suspension** (such as paints, rubber solution, or certain classes of crudes) is misleading, as, owing to the sluggish flow of the convection currents, the figure recorded by the thermometer by no means represents the temperature of the portion giving off vapour. Several means have been suggested for overcoming this difficulty. Redwood suggests the addition of a thermometer with a small cylindrical bulb fixed in a perpendicular position in the cover of the oil-cup, so that the bulb is only a tenth of an inch from the side of the cup. This has been adopted by the Indian Government for use with Burma Crude.

Some time ago, the writer designed a form of lid in

TABLE III.

TABLE FOR CORRECTION OF OBSERVED FLASHING-POINTS FOR VARIATIONS IN ATMOSPHERIC PRESSURE.

in ins.	27	27·2	27·4	27·6	27·8	28	28·2	28·4	28·6	28·8	29	29·2	29·4	29·6	29·8	30	30·2	30·4	30·6	30·8	31
Flashing-Point in Degrees Fahrenheit	60·2	60·5	60·8	61·2	61·5	61·8	62·1	62·4	62·8	63·1	63·4	63·7	64	64·4	64·7	65	65·3	65·6	66	66·3	66·6
	61·2	61·5	61·8	62·2	62·5	62·8	63·1	63·4	63·8	64·1	64·4	64·7	65	65·4	65·7	66	66·3	66·6	67	67·3	6
	62·2	62·5	62·8	63·2	63·5	63·8	64·1	64·4	64·8	65·1	65·4	65·7	66	66·4	66·7	67	67·3	67·6	68	68·3	68·6
	63·2	63·5	63·8	64·2	64·5	64·8	65·1	65·4	65·8	66·1	66·4	66·7	67	67·4	67·7	68	68·3	68·6	69	69·3	69·6
	64·2	64·5	64·8	65·2	65·5	65·8	66·1	66·4	66·8	67·1	67·4	67·7	68	68·4	68·7	69	69·3	69·6	70	70·3	70·6
	65·2	65·5	65·8	66·2	66·5	66·8	67·1	67·4	67·8	68·1	68·4	68·7	69	69·4	69·7	70	70·3	70·6	71	71·3	71·6
	66·2	66·5	66·8	67·2	67·5	67·8	68·1	68·4	68·8	69·1	69·4	69·7	70	70·4	70·7	71	71·3	71·6	72	72·3	72·6
	67·2	67·5	67·8	68·2	68·5	68·8	69·1	69·4	69·8	70·1	70·4	70·7	71	71·4	71·7	72	72·3	72·6	73	73·3	73·6
	68·2	68·5	68·8	69·2	69·5	69·8	70·1	70·4	70·8	71·1	71·4	71·7	72	72·4	72·7	73	73·3	73·6	74	74·3	74·6
	69·2	69·5	69·8	70·2	70·5	70·8	71·1	71·4	71·8	72·1	72·4	72·7	73	73·4	73·7	74	74·3	74·6	75	75·3	75·6
	70·2	70·5	70·8	71·2	71·5	71·8	72·1	72·4	72·8	73·1	73·4	73·7	74	74·4	74·7	75	75·3	75·6	76	76·3	76·6
	71·2	71·5	71·8	72·2	72·5	72·8	73·1	73·4	73·8	74·1	74·4	74·7	75	75·4	75·7	76	76·3	76·6	77	77·3	77·6
	72·2	72·5	72·8	73·2	73·5	73·8	74·1	74·4	74·8	75·1	75·4	75·7	76	76·4	76·7	77	77·3	77·6	78	78·3	78·6
	73·2	73·5	73·8	74·2	74·5	74·8	75·1	75·4	75·8	76·1	76·4	76·7	77	77·4	77·7	78	78·3	78·6	79	79·3	79·6
	74·2	74·5	74·8	75·2	75·5	75·8	76·1	76·4	76·8	77·1	77·4	77·7	78	78·4	78·7	79	79·3	79·6	80	80·3	80·6
	75·2	75·5	75·8	76·2	76·5	76·8	77·1	77·4	77·8	78·1	78·4	78·7	79	79·4	79·7	80	80·3	80·6	81	81·3	81·6

THOMSON AND REDWOOD'S "HANDBOOK ON PETROLEUM."

which the standard dimensions of the Abel Tester were adhered to, but the position usually occupied by the

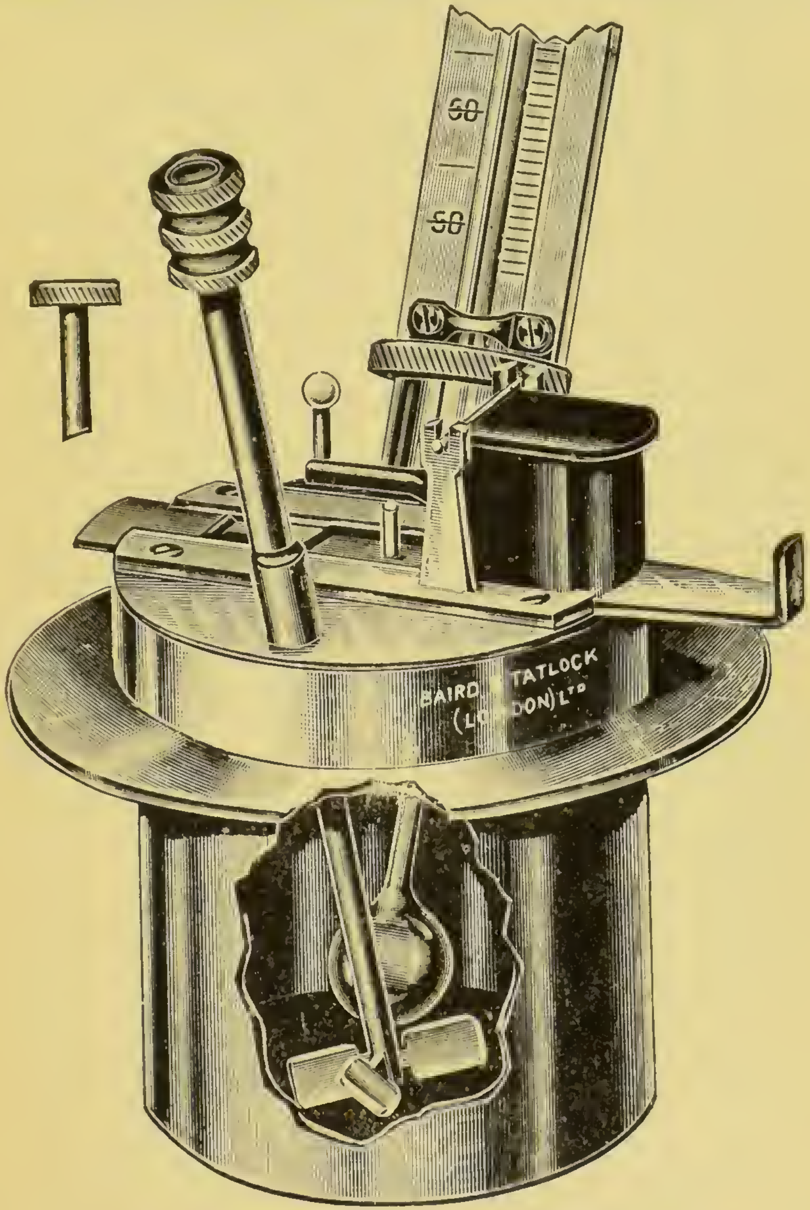


FIG. 8.—Section of modified Abel Petroleum Tester showing Plunging Agitator.

small ivory bead was taken by a plunging agitator and very satisfactory results have been uniformly obtained

with this modification. The problem has now been solved by the employment of rotating stirrers, and this solution is given legal recognition in this country by an Order in Council dated May 7, 1907 (Statutory Rules and Orders, 1907, No. 483), the schedule of which reads as follows :

Directions for Testing Petroleum Mixtures.

(1) **Liquid Mixtures.**—Where the petroleum mixture is wholly liquid, flows quite freely, and does not contain any sediment or thickening ingredient, such mixture shall be tested in the manner set forth in Schedule One to the Petroleum Act, 1879.

(2) **Viscous and Sedimentary Mixtures.**—Where the petroleum mixture contains an undissolved sediment, as in the case of some metal polishes, which can be separated by filtration or by settlement and decantation, the sediment may be so separated and the decanted liquid may be tested in the manner set forth in Schedule One to the Petroleum Act, 1879.

In carrying out such separation, care must be taken to minimise the evaporation of the petroleum. The separation of the sediment must not be effected by distillation.

Where the petroleum mixture is such that sediment cannot be separated by the aforementioned means, or where it is of a viscous nature, as in the case of india-rubber solution, quick-drying paints, &c., such mixture shall be tested in the apparatus modified as shown in the drawing hereto. This apparatus differs from that prescribed in Schedule One to the Petroleum Act, 1879, only in the addition of a stirrer to equalise the temperature throughout the sample under test.

In carrying out the test of a viscous petroleum mixture, this stirrer shall be constantly revolved at a slow speed, except when applying the test flame, with the fingers, the direction of revolution being that of the hand of a clock.

With the exception of the use of the stirrer, the manner of carrying out the test shall be that set forth in Schedule One to the Petroleum Act, 1879.

The stirrer may be removed by grasping the spindle just above the blades with the finger and thumb, and unscrewing the upper sheath. The opening in the lid, through which the stirrer passes, may then be closed by a plug provided for the purpose.

Fig. 9.—Abel-Pensky Petroleum Tester.

When this has been done, the apparatus shall be deemed to comply with the specification set forth in Schedule One of the Petroleum Act, 1879, and may be used for testing ordinary petroleum or solid petroleum mixtures.

A model of the aforementioned apparatus will be deposited with the Board of Trade, and the provisions of Section three of the Petroleum Act, 1879, in regard to verification and stamping shall apply also to such apparatus as though it were the apparatus prescribed by the said Act.

For the purpose of carrying out such verification the stirrer shall be removed and the opening plugged as hereinbefore directed. The apparatus shall then be tested with ordinary petroleum. The stirrer shall be verified by comparison of measurements.

For the examination of liquid samples that are in-

sufficient in bulk to fill the cup to the correct height, it is a useful procedure to pour what there is into the cup and then slowly to add water or mercury until the oil (which of course remains on the surface) reaches the required height.

Abel-Pensky.*—To avoid the personal error likely to be introduced in the opening of the slide of the Abel instrument, the German Government has adopted a clock-work movement for this operation. The test-flame is applied by pressing the trigger, and the mechanism is rewound after each test by turning the knob (Fig. 9). As used in Germany the Abel-Pensky gives a flashing-point some 3° F. higher than the Abel as standardised by the Board of Trade, but in India it is adjusted to give results in accordance with that apparatus.

Saybolt Electric Tester.—The use of this tester is confined almost entirely to the United States. It was adopted in 1879 by the New York Produce Exchange. The official directions for its use are as follow :

> " Fill the metal bath with water, leaving room for displacement by the glass cup. Heat the water until the bath thermometer indicates 100° F., at which point remove the lamp. Fill the glass cup with oil to the top line indicated by the rim surrounding cup, which is one-eighth of an inch below the top edge of the cup. See that there is no oil on the outside of the cup, nor upon the upper level edge, using paper to clean cup in preference to cotton or woollen material. See that the surface of the oil is free from air-bubbles before first flash is produced. Lift the cup steadily with left hand, and place in the bath. Suspend the thermometer with the bulb of same immersed just from view under the surface of oil. Adjust the flashing-bar and immerse the battery zincs in fluid. Try for

* Redwood, " Petroleum," p. 566.
Thomson and Redwood, " Handbook," p. 93.

first flash every degree until the same is obtained. Attain flash by producing spark with one stroke of the key. The stroke on the key should be such as in telegraphy is used to produce what is called a dot, that is, a short, quick stroke. The first flash produced from 110° test oil is generally obtained when the temperature of the oil has arrived at 90°. The temperature of the bath at

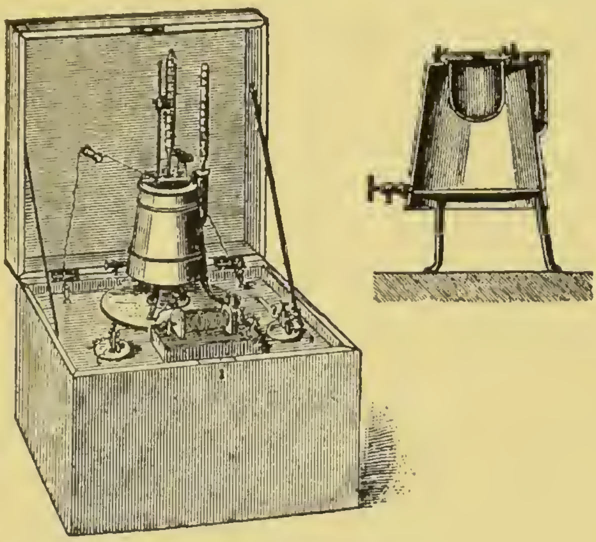


FIG. 10.—Saybolt Electric Tester.

100° (as per note above) will carry the oil to about 90°, or, in other words, to about the first flashing-point, without the aid of a lamp. When the thermometer in the oil indicates 90°, introduce lamp under the bath, and do not remove until the operation is finished.

"The temperature of oil when placed in bath should not be lower than 55°, nor higher than 70° F. The flashing-bar must be free from oil before adjusting for test. Draughts of air must be excluded from the apartment wherein tests are made. Oil of 110° and upwards shall (after first flash) be flashed at 95, 100, 104, 108, 110, 112, 115. Oil of 120° and upwards, after first flash, at 100, 105,

110, 115, 118, 120, 122, 125. Oil of 130° and upwards every 5° after first flash until burning-point."

It will be noticed that the results furnished are of the "open-test" and "fire-test" order.

Tagliabue's "Pyrometer." *—This apparatus for Open and Close Tests was patented in America in 1862 and is used as follows:

Remove the cover, and take out the oil-cup. Fill the water-bath with water to within 2 inches of the top. Replace oil-cup and fill with the oil to be tested to within ⅜ inch from the top, then put on the cover, and secure it in position by turning. Light the spirit-lamp under the bath and remove it when the thermometer reaches about 20° F. below the supposed flashing-point. When the lamp has been removed, press down the brass knob on the top of the cover, which will open the valves and admit air to the instrument and the vapour to the dome. Insert a very small lighted taper into the dome through the slot, and if the flash-point has been reached a slight puff will occur.

Failing this, replace the lamp and warm at the rate of 2° or 3° F. a minute, remove lamp, open valves, and apply taper until the "puff" is obtained. The reading of the thermometer at which this occurs is the flashing-point of the oil.

To ascertain the burning-point, replace the lamp, and, when the temperature has risen another 8° F., remove it, swing back the cover by the handle, and pass the lighted taper quickly across the oil; if the burning-point has been reached the oil will ignite. If not, replace the cover and the lamp and continue testing every 3° F. More than one testing of a sample should be performed, fresh oil and cold water being used, and all tests succeeding the first will probably

* Redwood, "Petroleum," p. 576.
Thomson and Redwood, "Handbook," p. 104.

give lower results, as the instrument can be more carefully watched.

The results furnished by the Tagliabue are not always satisfactorily concordant.

Elliot Tester.*—This is a modification of the Wisconsin State Tester, from which it differs chiefly in the substitution of a glass cover for a metal one. It was suggested by Professor Arthur Elliot, and was adopted by the State Board of Health of New York in 1882.

The Elliot Tester has a metal water-bath with a capacity of 20 fluid ounces, and a copper test-cup requiring 10 fluid ounces to fill it in the manner prescribed.

Above the cup proper is a vapour-space of larger diameter. The vapour-space is covered by a glass disc through which passes a thermometer held by a cork, and a hole $\frac{3}{4}$ inch wide for the insertion of a gas-jet $\frac{1}{4}$ inch long (a test flame of burning waxed twine may be employed). The oil-cup being removed, the water-bath is filled to a mark and the cup is replaced. Oil is poured into the cup to a point $\frac{1}{8}$ inch below the flange between the oil-chamber and the air-space, without allowing the walls of the latter to become oily. The glass cover is placed in position with the bulb of the thermometer just covered by the oil. A small Bunsen or spirit-lamp is lighted underneath, and the rate of heating regulated to 2° F. per minute.

The flash-torch is introduced at every 2° rise, to about half-way between the oil and the cover, and this is done from 85° to 95° F., when the lamp is removed and the testing continued at every degree to 100° F. After that point the lamp is replaced and the test applied every 2° F.

The cover may be removed and the thermometer suspended in the oil in order to ascertain the fire test of a sample. In this case the rate of heating is not to exceed 10° F. a minute.

* Redwood, "Petroleum," p. 577.
Thomson and Redwood, "Handbook," p. 107.

Foster Automatic Tester.*—In Ohio an apparatus is used in which a wick dipping into the sample gives a small flame constantly burning at an opening in the cover of the cup, and the flashing-point is indicated by the extinction of this flame by the slight explosion occurring. The oil-cup is exactly filled to a gauge-mark, the water-bath is half filled, the rate of heating is 2° F. a minute, and the wick is lit at 100° F.

Granier Tester.†—This is another "automatic" tester, on somewhat similar lines to the Foster, with the addition that the oil is heated by a copper wire passing from the test flame into it.

With both these automatic testers the results obtained are so unsatisfactory that lengthened directions for use would not be warranted.

Open Test and Fire Test of Kerosene.—This may be ascertained by a modified use of the Abel instrument, although it is preferable to employ the usually unavailable apparatus which was legalised here in 1868 until supplanted by the Abel. The rate of heating may be two degrees a minute, and the application of the test flame at every degree similar to that of the lubricating oil test.

The foregoing instruments are of little use for **temperatures over 150° F.** And, owing to the employment of soft solder in the construction of some of them, a word of warning is necessary against their being heated at all strongly. To the knowledge of the writer an Abel cup has been wrecked in this way by more than one careless operator.

The following are designed for use with **heavier oils.**

Pensky-Martens Tester.‡—The apparatus most generally employed for oils of high flash-point is the

* Thomson and Redwood, "Handbook," p. 112.

† *Ibid.* p. 116.

‡ Redwood, "Petroleum," p. 593.
Thomson and Redwood, "Handbook," p. 124.
Archbutt and Deeley, "Lubrication and Lubricants," p. 211 (ed. 1912).

Pensky-Martens instrument. The cup is of the same dimensions as that of the Abel, and the lid has an arrangement for the application of the test flame in a similar manner to the one adopted with that instrument, but a modification enables the test to be applied by turning a non-conducting button. Besides which, stirrers are provided to ensure uniform temperature of the contents. Before determining the closed test, the cup and lid with the attached stirrers are very thoroughly cleansed from any oil remaining from a previous sample. By forcing the manipulating button upwards, the revolving plate on the cover may be removed, and the cleaning facilitated. If the oil last under examination was of a much more volatile nature than the sample in question, it may be well to reject the result of the first test made with the heavier oil, and to use this experiment as a means of completely freeing the cup from any foreign vapour.

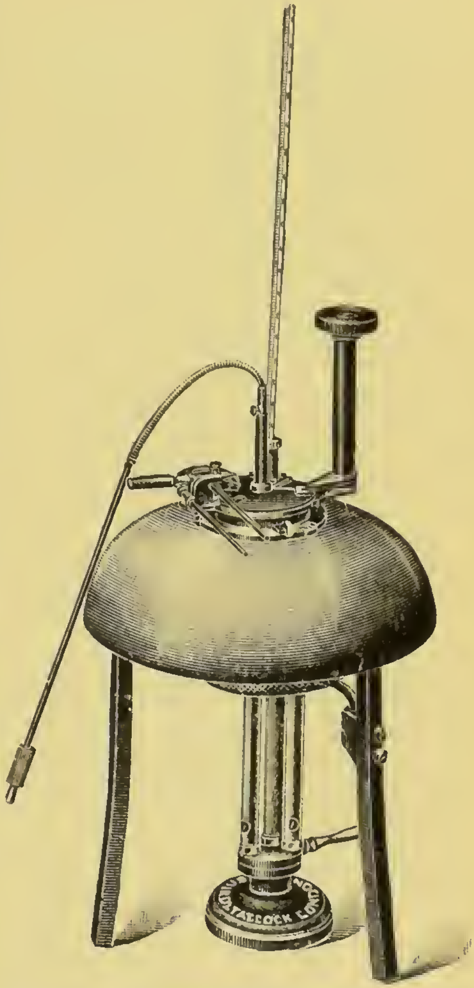


FIG. 11.—Pensky-Martens Tester.

When thoroughly cleansed the cup must be filled to the line inscribed, the lid put on so that it is well "home," and the cup placed in the air-bath. The heating, by means of a Bunsen under the air-bath for

at least 50° below the flashing-point, is to be at the rate of 10° a minute, and the test is to be applied at

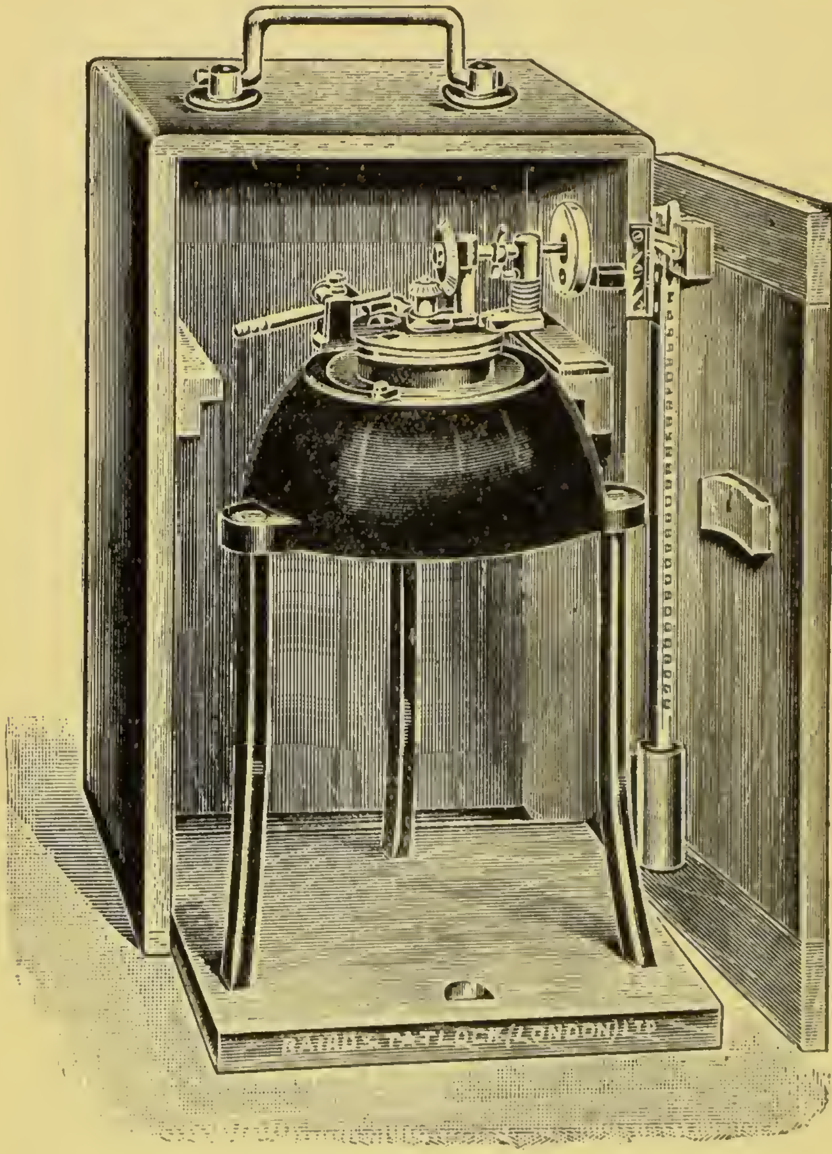


FIG. 12.—Gray's Tester.

every 2° F. rise during the same time as the Abel Tester. During the whole experiment the stirrers must be kept revolving with a steady, continuous motion, but during each actual application of the test

flame it is usual to cease agitation. Care should be taken that in stirring none of the oil is thrown up on to the lid of the cup, but no difficulty need be experienced here if the stirring be neither jerky nor too rapid.

A nitrogen pressure thermometer, reading to 700° F., is useful for cylinder oils, but for other lubricants an ordinary mercury thermometer reading to 550° F. usually suffices. The results furnished by the Pensky-Martens Tester approximate very closely to those given by the Abel Tester at those temperatures at which it is possible to use either apparatus.

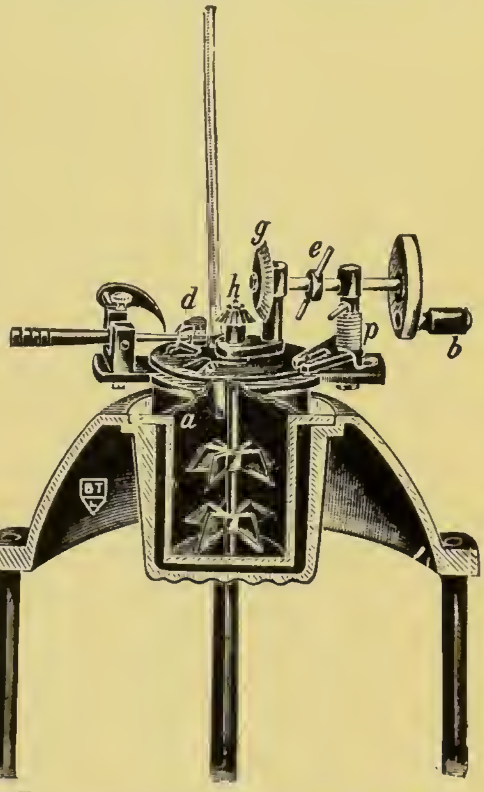


FIG. 13.—Section of Gray's Tester.

Gray's Tester.* —A form of heavy-oil tester closely resembling the Pensky-Martens is the Gray.

The chief variation consists in the means for rotating the stirring-vanes and for applying the test flame. Both these operations are performed from a non-conducting button fixed on the end of a horizontal shaft, which also carries one of a pair of bevelled wheels, and is pierced by a short pin. The other bevelled wheel is fixed on the upper end of the spindle to which the vanes are attached, and by turning the button

* Redwood, "Petroleum," p. 595.
Thomson and Redwood, "Handbook," p. 127.
Archbutt and Deeley, "Lubrication and Lubricants," p. 213.

slowly by means of its handle the oil is kept gently agitated.

In making a test, the horizontal shaft is slid back (a certain amount of "play" being allowed it by the supports) until the pin engages with a projection attached to the sliding cover of the lid, then, by grasping the button itself and turning it firmly, the cover is opened.

The same general remarks apply to the Gray Tester as to the Pensky-Martens, and the results furnished by the Gray should be concordant with those obtained with the other apparatus.

Open Test and Fire Test of Heavy Oils.—These tests, in conjunction with the Close Test, give some idea as to the **homogeneous character of the sample** under examination.

It is possible, for instance, to detect the presence of a small percentage of a light oil in a lubricant by the wide range covered by the three tests. Very often, in such cases, if the proportion of light oil be not too great, by allowing the cupful to cool after the fire test is taken and then repeating the whole experiment, a very great difference between the first and second close tests, a smaller difference between the open tests, and a practical agreement between the fire tests will confirm such an inference. But it must always be remembered that this treatment will invariably cause *some* alteration in the lower figures.

The usual way of taking the open and fire tests is to continue the heating when the close test has been ascertained, and, removing the lid of the cup, to hang the thermometer from a retort stand with its bulb immersed about a quarter of an inch below the surface of the oil. Where absolute accuracy is needed, a fresh portion of oil should be taken, and the open test and fire test made with the lid of the cup removed throughout the experiment, and the level of the oil at the temperatures of the results not much more than an eighth of an inch below the top of the cup. At every

2° F. a test-flame of gas, $\frac{3}{16}$ of an inch in diameter, is passed across the surface of the oil slightly below the level of the cup, taking care that it does not come in contact with the oil. The heating remains at the same rate of 10° F. a minute. That point at which a flicker of flame covers the *whole* of the surface is noted as the open flash, and the fire test is the temperature at which the oil vapours continue to burn until the next application of the flames is due.

The two higher tests may also be performed by heating the oil in a porcelain crucible of about two inches diameter in a sand-bath. The crucible is filled to about a quarter of an inch from the top, and the level of the sand and oil should be in the same plane.

In the absence of coal-gas for the test flame a useful substitute can be provided by hydrogen or air which has been passed through a Woulff's bottle containing cotton wool saturated with light petroleum spirit.

CHAPTER III.

VISCOSITY.*

THE "body" or viscosity of an oil is most commonly measured by its rate of flow through an orifice of certain dimensions. The results obtained are, of course, entirely comparative, and they are only of use as a guide in the valuation of lubricants when compared with those given by samples of the same nature and of known value for any specific purpose.

Redwood Viscometer.†—The form of viscometer adopted almost universally in this country is that known as the Redwood.

The instrument consists of a silvered brass oil-cylinder, furnished with an agate jet, and surrounded by a copper bath. A copper tube, closed at the lower end, projecting at an angle of 45° from the side of the bath near the bottom, provides a means of heating the bath liquid, and by the use of a revolving agitator, which forms part of the apparatus, the heated liquid rising from the copper tube can be uniformly distributed through the bath. But for temperatures not far removed from that of the room in which the work is being done, constancy is most easily attained by the addition of small quantities of water heated in a small beaker.

The agitator carries a thermometer, to indicate the temperature of the bath. The oil-cylinder is furnished

* Archbutt and Deeley, "Lubrication and Lubricants." Redwood, "Petroleum," p. 597, 614.

† Redwood, *J. Soc. Chem. Ind.*, March 1886, "Petroleum," p. 600.

(*For Redwood Viscometer, Admiralty Pattern, see p. 70.*)

with a stopper, consisting of a small brass sphere attached to a wire, the sphere resting in a hemispherical cavity in the agate jet. A short standard attached to the oil-cylinder carries a clip to support a thermometer in the oil. The diameter of the *stem* of this thermometer must be a quarter of an inch. Inside the oil-cylinder, and at a short distance from the top, is fixed a small bracket, terminating in an upturned point, which forms a gauge of the height of the oil-level. The instrument is supported on a tripod stand provided with levelling screws.

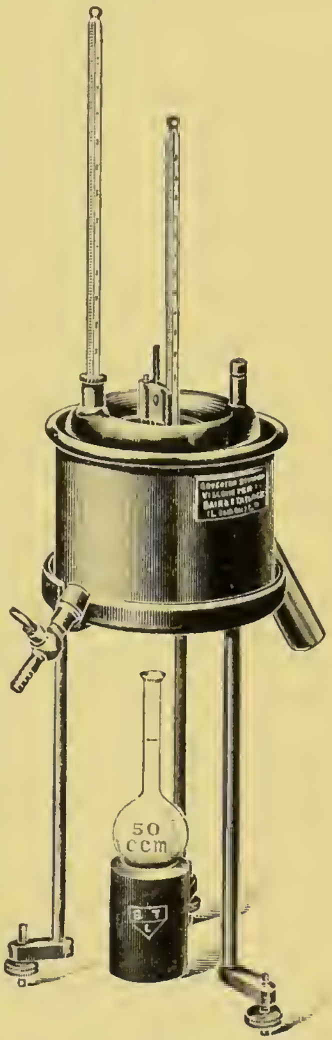


FIG. 14.—Redwood Viscometer.

The bath is filled with a suitable liquid to a height roughly corresponding with the point of the gauge in the oil-cylinder. Water answers well for temperatures up to 200° F., and for higher temperatures a heavy mineral oil may be used. The liquid having been brought to a temperature slightly in excess of that at which the viscosity is required, the oil to be tested is poured into the oil-cylinder until the level of the liquid is above the point of the gauge. When the inner thermometer remains steady at the position required, the level of the oil is adjusted to the height of the gauge-point, and a narrow-necked flask, holding 50 c.c. to a point marked on the neck, is placed beneath the jet in a vessel containing a liquid of the same temperature as the oil. The ball-valve is then raised, a stop-watch

at the same time started, and the number of seconds occupied in the outflow of 50 c.c. noted. It is of the greatest importance that the oil-cylinder should be filled exactly to the point of the gauge after inserting the thermometer, and that the given temperature should be precisely maintained during experiment, a difference of ½° F. making an appreciable alteration in the viscosity of some oils. It is also essential that the oil should be quite free from dirt or other suspended matter, and from globules of water, as the jet may be partially obstructed by them. If the oil-cylinder requires to be wiped out, paper rather than cloth should be employed, as filaments of the latter may be left adhering. When oils are being tested at temperatures much above that of the laboratory a gas flame is applied to the copper heating-tube, and the agitator kept in gentle motion throughout the experiment. The jet should be carefully examined before the apparatus is used, and, if necessary, should be cleansed by passing a piece of soft string through it.

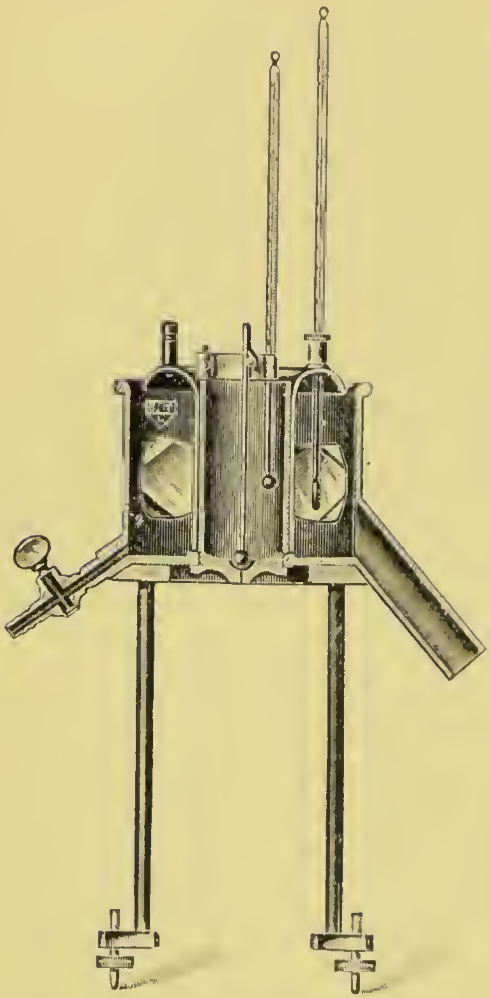


FIG. 15.—Section of the Redwood Viscometer.

The apparatus should be adjusted by means of the levelling screws, so that a spirit-level placed on the top of the oil-cup shows it to be horizontal.

The viscosities at 70° and 140° F. are usually sufficient for ordinary lubricating oils, and for cylinder oils those at 200° and 250° F.

The results are generally stated in terms of Rape Oil at 60° F., this being taken as equal to 100.

When the instrument was first designed, a large number of rape oils were tested in it, and the average of these tests was found to give 535 seconds. Therefore the number of seconds obtained must be multiplied by 100 and divided by 535*. In addition, a correction is to be made for the influence of the different specific gravities on the rate of outflow, and the figure obtained from the division by 535 is therefore to be multiplied by the specific gravity of the oil at the temperature of the experiment and divided by ·915 (Sp. Gr. of Rape Oil at 60° = ·915).† The result will give the viscosity in terms of Rape Oil at 60° F. multiplied by 100.

The Redwood Viscometer requires about 6 fluid ounces of oil for the performance of a test.

Owing to the peculiar "lag" in the complete alteration of viscosity shown by many viscous oils when heated or cooled, the sample for examination should

* The rape oil of the present day does not, as a rule, give a figure so high as this, but, for the sake of uniformity, the original figure 535 is adhered to.

† The most rapid method of calculating the viscosity in terms of rape oil is to multiply the number of seconds by the specific gravity at the temperature of the experiment, and to divide the result by 489·525 (= 535 × ·915). For the purpose of this division a copy of the following table will be useful :

489·525	×	1	=	489·525
489·525	×	2	=	979·050
489·525	×	3	=	1468·575
489·525	×	4	=	1958·100
489·525	×	5	=	2447·625
489·525	×	6	=	2937·150
489·525	×	7	=	3426·675
489·525	×	8	=	3916·200
489·525	×	9	=	4405·725

be kept for twenty-four hours at the temperature of working when this is much below 100° F.

For very exact determinations it is recommended that the test should be performed in a cupboard, at the same temperature as the viscometer whenever this is possible, thus overcoming the otherwise serious difficulty introduced by the cooling of the lower part of the agate. When the oil is very fluid this drawback is not felt so much, as the rapidity of the stream keeps it at a more uniform temperature. In those cases where it is necessary to keep a burner under the heating arm throughout the test, care should be taken that the hot gases from the flame do not impinge on the under side of the agate.

Some misunderstanding has been caused by the mention of 25·5 seconds as the time taken for the outflow of 50 c.c. of distilled water at 60° F. It has been found in the manufacture of the Redwood Viscometer that it is practically impossible to drill a jet which will exactly give this figure with water, and which will at the same time give a result with more viscous liquids, agreeing with that obtained with the original apparatus. So that, although 25·5 seconds was shown by distilled water in the original, it is considered better to neglect this when the viscometers are standardised, and to rely only on the results yielded by different oils.

Engler Viscometer.*—In the Engler instrument the jet is a metal tube, and the water- or oil-bath is carried underneath the oil-cup with the object of keeping the whole length of this tube at the same temperature as the rest of the apparatus. The oil-cup is covered by a loosely fitting lid, and the jet is closed by a plug of hard wood passing through this lid, so that it can be opened without uncovering the oil. A ring-burner under the bath serves for heating.

To standardise, the water-bath is filled with water

* Redwood, "Petroleum," p. 602.
Archbutt and Deeley, "Lubrication and Lubricants," p. 173.

and warmed to 20° C., the oil-cup is filled with distilled water to the height of the three points, and is levelled by placing pieces of card under one or more of the three feet until each point is just immersed in the liquid. The temperature as indicated by both thermometers is brought to 20° C. and the time noted for the outflow of 200 c.c. The experiment is completed three times and the average of the three results, which should not differ by more than half a second, is taken. If the apparatus is correctly made, the mean will be between 50 and 53 seconds. The whole number nearest to this average is taken as the basis of future calculations.

The oil to be tested is poured into the inner cup until level with the points, and stirred until the thermometer indicates the required temperature. The lid is then put on and 200 c.c. are run into the measuring flask.*

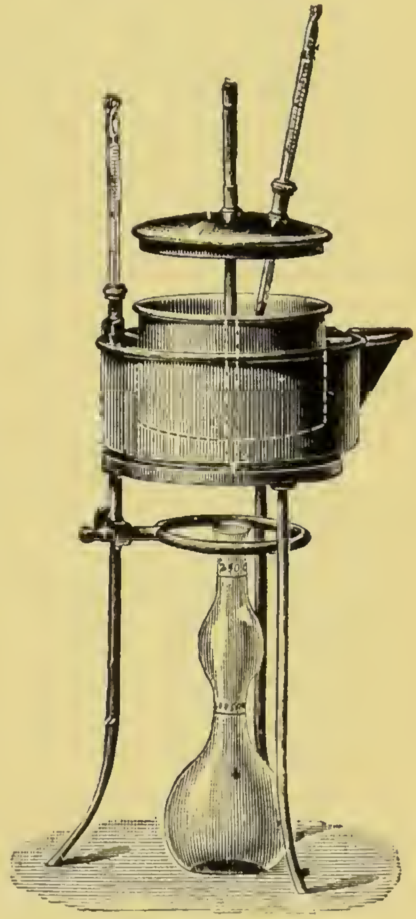


Fig. 16.—Engler Viscometer.

The test should be repeated, and two results should not differ by more than 1 per cent. The resulting average number of seconds is divided by the seconds taken by water, and the quotient gives what Engler calls the "specific viscosity" of the sample at the temperature of the experiment. The flask may be

* The 240 c.c. mark on the measuring flask, which is supplied, shows the capacity of the oil-cup to the height of the points.

placed in a bath at the same temperature as the oil-cup, to avoid the error due to cooling.

The Engler Viscometer has been found in practice to show roughly about 170 seconds to be equivalent to 100 seconds in the Redwood. 240 c.c., which the Engler Viscometer requires for working, are equal to nearly 8½ fluid ounces.

Engler-Kunkler Viscometer.*—Owing to the absence of stirrers in the Engler Viscometer, variations in temperature during the experiment are difficult to avoid, particularly at high temperatures. To meet this drawback, Engler and Kunkler have designed an envelope of metal which covers the entire apparatus, and can be heated by a flame placed underneath, thus forming an oven, which is so constructed that the experiment can be conducted from the exterior without opening.

To operate, the flask is placed in the oven on the stand below the plate holding the viscometer, then the plate and viscometer are also put in position, and the cover of the oven is firmly fixed. (The marks on the plate, viscometer, and cover must agree in position with that on the side of the oven.) Next, the thermometers are inserted, the outer being on a level with the oil-cup and the inner at the bottom of it, and the tube for filling is put in so that it dips into the lip of the oil-cup. The stirrer is lowered into the cup, and the whole apparatus levelled by the plumb-line at the side. The oven is heated until the outer thermometer registers the required temperature. While this temperature is being reached, the can supplied with the apparatus is filled with the sample and heated to half a degree or so above the requisite temperature, the contents being stirred meanwhile. The can being then full to the height of the indicator, the oil is quickly poured into the viscometer through the filling tube until all the points in the viscometer are just covered.

* Redwood, "Petroleum," p. 605.
Archbutt and Deeley, "Lubrication and Lubricants," p. 175.

The stirrers are worked backwards and forwards, and as soon as the temperature is constantly correct they are lifted out of the oil and the plug withdrawn.

The hole in the cover left by the plug should be closed with a cork.

Saybolt Viscometer.*—A form of viscometer employed in the United States is that known as the Saybolt. It consists of a water-bath of large capacity and an oil-cup holding less than the Redwood or the Engler instrument.

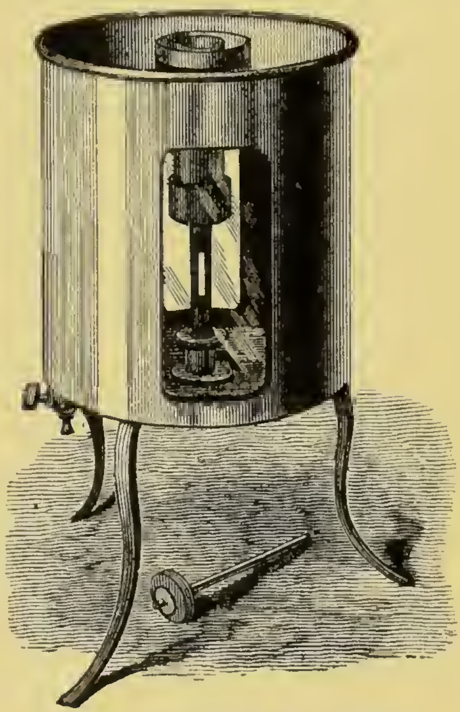


Fig. 17.—Saybolt Viscometer.

The jet is of metal. As the volume of liquid flowing out is measured while in the oil-cup no error due to contraction in a cool external measuring flask can arise.

In use, the outside bath is filled with water at the required temperature (there is no arrangement for heating), and the sample to be examined is poured into the cup until it overflows into the gallery at the top, a cork being inserted into the tube at the bottom of the apparatus into which the jet delivers. The oil is stirred with a thermometer, and when at the right temperature the thermometer is removed, the displaced oil returning to the cup from the gallery. All surplus oil in the gallery is then extracted with a pipette. The cork is removed from the bottom tube at the same time as a stop-watch is started, and the watch is stopped on the

* Redwood, "Petroleum," p. 604.
Archbutt and Deeley, "Lubrication and Lubricants," p. 172.

surface of the oil appearing at the window of the oil-cup.

An oil showing 100 seconds for 50 c.c. in the Redwood Viscometer should show roughly about 56 seconds in the Saybolt when tested in the manner described. The Saybolt Viscometer requires nearly three fluid ounces for operation.

Glass Jet Viscometers.—The three viscometers already described are the best-known forms of jet viscometers which had their forerunners in ordinary glass pipettes that were filled to a mark and emptied to a mark. These primitive instruments were unsatisfactory for many reasons, but especially because only those results obtained with the same pipette were comparable, and only very rough attempts were made to regulate their temperature; moreover they were liable to fracture.

Coleman-Archbutt Viscometer.*—The least unsatisfactory form of glass viscometer, perhaps, is Archbutt's improved form of the viscometer first used by Coleman about 1869. It consists of a glass pipette, contained in an outer water-jacket with a funnel for pouring in hot or cold water, a tube for running off water, and a stirrer for thoroughly mixing the water in the jacket. The neck at the lower end of the jacket is made narrow, a rubber stopper not more than $\frac{3}{16}$ inch thick is fixed in it, and the jet of the efflux tube projects through the stopper only $\frac{3}{16}$ inch, and does not extend quite to the end of the neck. Thus the oil in the tube is surrounded by the water in the jacket until it has reached nearly to the end of the jet, and the temperature is maintained constant until the oil has passed out of the tube. The jet is protected from change of temperature as well as from fracture by being contained entirely within the neck of the jacket. The efflux tube is narrowed for a short distance above the jet, and four circumferential

* Archbutt and Deeley, "Lubrication and Lubricants," p. 169.

marks are etched upon it. The lowest or zero mark is rather above the middle of the narrow portion ; the

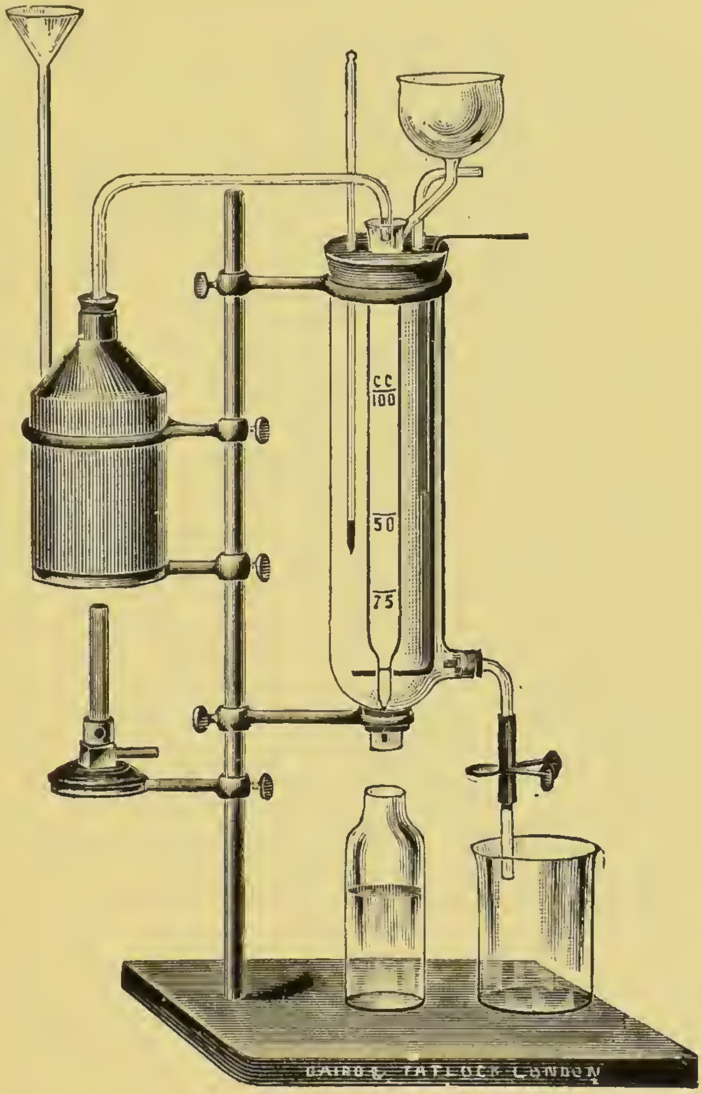


FIG. 18.—Coleman-Archbutt Viscometer.

other three marks are on the wide portion, and divide the tube above the zero mark into capacities of 25 c.c., 50 c.c., and 100 c.c. The volume of oil used for a test may therefore be varied according to the viscosity and

the quantity available. But the tube must be separately standardised, from each mark, as the times of efflux of the different volumes bear no simple relation to each other. The temperature is maintained constant by pouring hot or cold water into the jacket, through the funnel, and running off the excess as often as required, using the stirrer frequently. The temperature is indicated by a thermometer immersed in the water. The oil, having been brought to about the desired temperature, is poured into the efflux tube, where the final adjustment is made by stirring with a thermometer, which is removed before the oil is run out. The jet is closed by a plug of soft wood, which is now removed and replaced by the finger, and the level of the oil is adjusted exactly to the mark which it is desired to run it from. It is then allowed to flow out, and the time occupied in reaching the zero mark is measured by a stop-watch, and compared with the time occupied by the standard oil to flow out under exactly similar circumstances. When it is desired to make a determination at 212° F., the temperature of the water is gradually raised to about 180° F., by pouring in very hot water, and then the funnel is removed and replaced by a tube connected with the metal boiler, and steam is blown in until the water boils. A short bent glass tube is provided for the escape of steam. The length of the jet is about 1 inch, and the diameter is such that 100 c.c. pure rape oil at 60° F. take about ten minutes to flow out.

Thurston's Oil Tester.*—Efforts have been made to reproduce in the laboratory the actual conditions under which the lubricant is used in practice. But all such methods of testing are of somewhat doubtful value, inasmuch as the bearings are in such perfect condition that the requirements of practice are not fulfilled.

Such a machine as that of Thurston, the best known of the type, requires power to drive it, and it must be

* Redwood, "Petroleum," p. 621.

possible so to control the power used as to regulate the speed within any required limit. A measured quantity of the oil is introduced into the journal and the load under which the oil is to be used applied by means of the spring in the pendulum. The spindle being

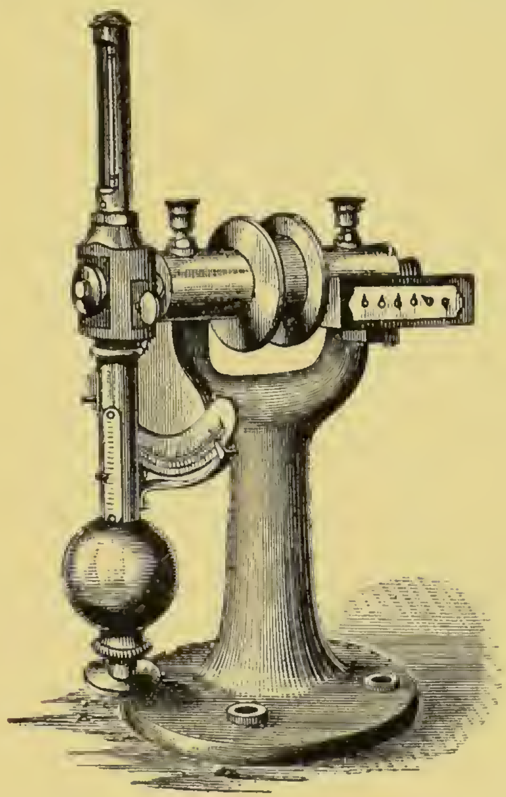


Fig. 19.—Thurston's Oil Tester.

revolved at the required speed, the friction-reducing power of the oil is inversely measured by the angle through which the pendulum is moved. The rise of temperature as indicated by the thermometer during a given period affords a further indication of the value of the sample.

Doolittle's Torsion Viscometer.*—Yet another principle is embodied in the Torsion Viscometer, namely,

* Redwood, "Petroleum," p. 612.

the retarding effect of the oil between two surfaces, one of which is fixed, the other having had given to it a rhythmical motion. The needle attached to the wire is set to the zero of the circular scale, and the wire is twisted through exactly 360° and released. The reading at the end of the first " swing " is noted, the next stop is ignored, and the third stop again noted. The whole operation is then repeated, reversing the direction of the twist, and the average difference between the first and the third stops gives the retardation due to the " body " of the sample.

CHAPTER IV.

COLOUR.

THE general appearance of mineral oils has a decided influence on their sale, and although a light or dark colour is to a great extent a matter of prejudice or sentiment, the analyst is very often called on to state the character of the sample in this respect.

The colour of **burning oils** is commercially taken with the Wilson Chromometer and that of **lubricants** with the Lovibond Tintometer.

Wilson's Chromometer.*—This instrument consists of two similar tubes, 16 inches in length, closed at each end by a screw cap carrying a stout glass disc. Light is reflected upwards through the tubes by a mirror, then twice reflected by two pairs of prisms, and thus brought into an eyepiece. One of the tubes is filled with the oil to be tested, and beneath the other, which is empty, a disc of stained glass is placed.

On looking through the eyepiece, the field is seen to be divided by a sharp line formed at the juncture of the two pairs of prisms, the two halves of the field being tinted respectively with the colour of the oil and that of the Standard. An accurate comparison can thus be made. The four Standards used are known as "Water White," "Superfine White," "Prime White," and "Standard White"; and glasses of these degrees of colour prepared from Mr. Robert Redwood's Standards are provided with the apparatus. A third tube similar

* Redwood, "Petroleum," p. 544.

to the other two is useful in order to directly compare two samples with each other, without making the tube which holds the standard glass oily.

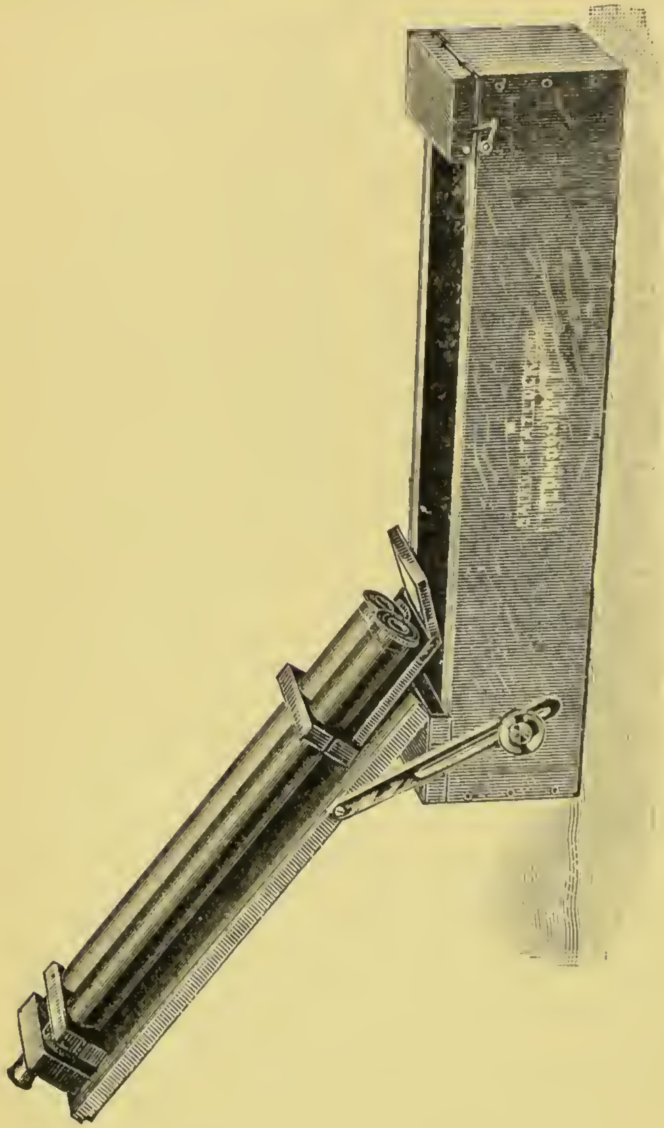


FIG. 20.—Wilson's Chromometer.

Lovibond's Tintometer.*—The 2-inch cell is filled with the sample which has been cleared by slightly warming (a high temperature will darken the oil), and is placed in one side of the tintometer. Standard glasses

* Redwood, "Petroleum," p. 592.

of Mr. Lovibond's series "500" are then put in the slots provided in the other side until a match is obtained with light reflected from the milk-glass reflector when viewed from the eyepiece. It is advisable to repeat the experiment by reversing the sides for the sample and glasses, and the two readings thus obtained should agree to within 1 or 2 per cent.

Stammer's Colorimeter.*—This is largely used on the Continent for the colour-measurement of oils, and

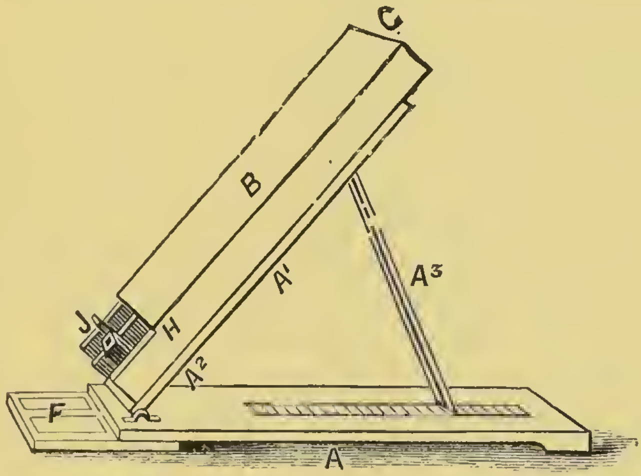


FIG. 21.—Lovibond's Tintometer.

has the advantage of showing from one standard the exact length of the column of liquid that matches it. The tube for the sample is depressed by means of the thumbscrew to its lowest position and removed for filling; it is then replaced. The box containing the prisms (similar to those of the Wilson and having the same effect) is lifted off, the standard glass is placed in the cell in the left-hand tube, and the box is replaced. On now looking through the eyepiece and adjusting the mirror at the base, a similar view is obtained to that in the Wilson. The thumbscrew at the rear of the apparatus is then turned until the two halves of the

* Redwood, "Petroleum," p. 544.

field appear of equal colour, and the reading on the

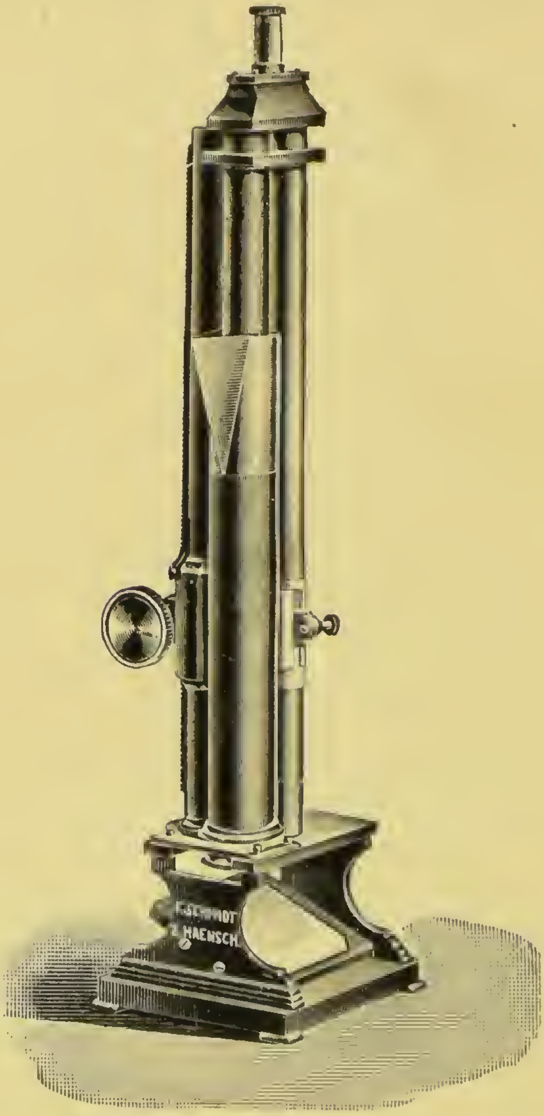


FIG. 22.—Stammer's Colorimeter.

scale in millimetres is recorded as the colour of the oil.

CHAPTER V.

SUNDRY APPARATUS.

Boiling-Point of Petrol, &c.—100 c.c. of the sample are measured into a Wurtz flask of the following dimensions:

Diameter of bulb .	$2\frac{3}{4}$ inches.
Diameter of neck .	$\frac{3}{4}$ inch.
Exit tube . . .	$2\frac{1}{2}$ inches from shoulder.

The flask is connected to a Liebig condenser 24 inches long, and is supported on a sand dish heated by a Bunsen burner. A thermometer with a sensitive bulb is inserted not too firmly, through a cork in the neck of the flask. The 100 c.c. cylinder used for measuring the sample stands close under the open end of the condenser, the junction with which may be wrapped round with a piece of paper to minimise loss by evaporation. The heating of the flask is so regulated that a decidedly broken stream of distillate falls from the end of the condenser. Before making a test of the percentage distilling below any given temperature, or before the first of a series of such tests, a few c.c. of spirit should be distilled through the apparatus to eliminate the error otherwise caused by liquid remaining in the condenser tube.

Initial Boiling-Point.—The thermometer is at first adjusted with its bulb half immersed in the liquid, but when ebullition becomes general and, with a good light, a layer of vapour can be seen steadily rising in the flask, it is steadily raised so that its bulb is just

covered by the vapour until the first drop of condensed spirit falls from the end of the delivery tube into the condenser. The temperature then shown by the thermometer is recorded as the initial boiling-point.

Final Boiling-Point.—The temperature shown by the thermometer when the bottom of the flask first becomes free of liquid.

Fig. 23.—Apparatus for testing the Pressure of Petroleum Vapour.

Pressure of Naphtha in Closed Vessels.*—The high coefficient of expansion possessed by petroleum spirit and its high vapour-tension may generate considerable pressure in its containing vessel on a slight rise in temperature. It is often necessary that an idea should be formed as to the amount of strain likely to be imposed on such vessels by their contents in order that a sufficient factor of safety may be allowed in their strength. The method devised for this purpose by Captain Thomson (His Majesty's late Chief Inspector of Explosives) and Sir Boverton Redwood involves the employment of a glass tube 6 inches long by 1 inch in diameter joined at its upper end to a short piece of $\frac{1}{4}$-inch tube, and at its lower end to a capillary tube, which is bent up to a height of some 30 inches.

On to the short tube is firmly wired a short piece of rubber pressure-tubing, which can be closed with a screw clip close to the glass. Mercury is poured into the 1-inch tube up to the mark near the bottom, and nine-tenths of the remaining space is filled with the sample to be tested. (The upper mark shows the correct height.)

The tube is then placed vertically into water maintained at 50° F., and the level of the spirit exactly

* Thomson and Redwood, "Handbook," p. 130.

adjusted when the temperature is constant. The screw clip is then firmly closed, and the apparatus is plunged into another bath of water which has been previously heated to 100° F.* The highest position attained by the mercury in the capillary tube is marked, and its vertical distance above the lower mark of the 1-inch tube recorded. The temperature of the bath is maintained at 100° F. for half an hour, and then the height of the mercury is again noted. This second reading should not exceed 24 inches if the spirit is to be contained in the vessels recommended.

Detection of Petroleum Vapour.†—In view of the enormous volume of mineral oil products stored and conveyed every year, the number of men engaged in handling it, and the costly nature of the installations and vehicles employed, the use of every possible precaution on the part of those responsible becomes a primary duty.

In many cases this care by no means ceases when the oil has passed out of hand, for with the lighter liquids, naphtha and kerosene, their evolved vapours remain for some considerable time after their removal, so that before any fire can be allowed to approach the empty spaces for cleaning, repairs, or other purposes, means must be taken to prove the perfect dispersion of such gases.

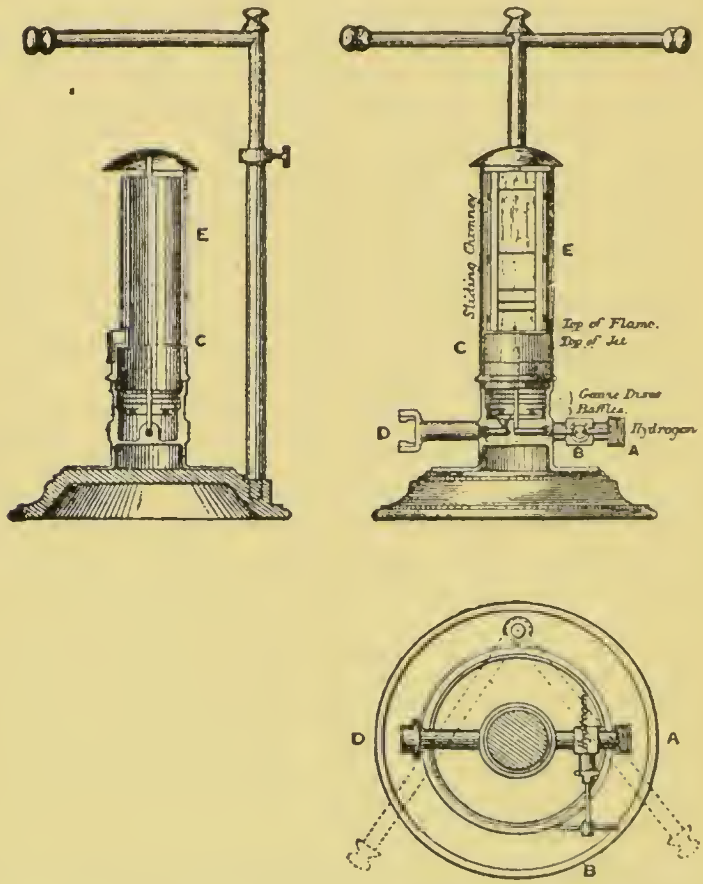
Moreover, the same precautions are, of course, often required in confined spaces adjacent to such liquids, into which the vapours may have found their way.

The mode of testing most commonly adopted is that known as the Clowes-Redwood system, and depends on the occurrence of a "cap" of ghostly appearance over a non-luminous flame burning in the contaminated atmosphere.

* If the water be well above the top of the rubber tubing any leakage will be visible.

† Clowes and Redwood, "Detection of Inflammable Vapour," (Crosby Lockwood & Co.)
Redwood, "Petroleum," p. 588.
Thomson and Redwood, "Handbook," p. 17.

In a paper on "The Transport of Petroleum in Bulk," read before the Institute of Civil Engineers (*Pro-*



FIGS. 24 and 25.—Apparatus for detecting Petroleum Vapour.

ceedings, CXVI. (1893–4), Part II.), Sir Boverton Redwood thus described the working of this test :

> " In the use of the apparatus the first step is to connect the hydrogen cylinder with the lamp, taking care that the unions are screwed up gas-tight. The sliding chimney of the lamp being raised about half-way, the gas is then cautiously turned on at the cylinder, the regulating valve on the lamp being left open, and a light is applied to

the hydrogen jet. The valve on the hydrogen cylinder is then adjusted so as to give a flame rather more than 10 millimetres (0·4 inch) in

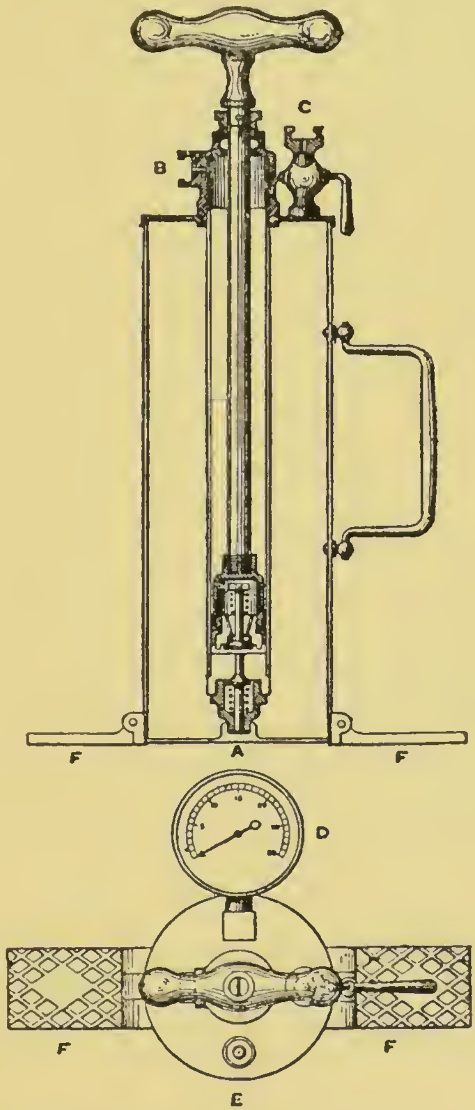


FIG. 26.—Apparatus for detecting Petroleum Vapour.

length, and the lamp chimney pushed down until there is an opening of only about a quarter of an inch in height at the bottom. This opening is left

for the supply of air to the hydrogen flame during the few minutes occupied in the warming of the chimney. As soon as the moisture which at first condensed upon the cold glass has evaporated the lamp is ready for use, and, assuming the collecting vessel to have been already charged with the sample to be tested, and connected with the lamp, all that remains is for the observer to completely close the sliding chimney of the lamp, adjust the hydrogen flame by means of the regulating valve on the lamp, so that the tip of the flame is only just hidden when the eye of the observer is on a level with the bottom of the window, place his head under a cloth such as used by photographers, so as to exclude light, and, as soon as his eyes have become sufficiently sensitive, turn on the tap of the collecting cylinder, and carefully observe what takes place in the lamp chimney.

"The tap may at once be turned on fully, as the construction of the outlet and inlet orifices prevents the sudden rushing out of the contents of the cylinder, and the sample will be gradually delivered into the test lamp during a period of more than two minutes, which is ample time for noting the effect. The rate of delivery is, of course, a gradually diminishing one, but this is not found to be attended with any inconvenience, the conditions being the same in each experiment.

"In this way a proportion of vapour, considerably below that which is required even for the production of an inflammable mixture, and still lower than that which is needed to give an explosive atmosphere, may be detected by the formation of a flame-cap of greyish blue colour, which, though faint, is easily seen, especially after a little practice.

"With an increase in the quantity of vapour, the flame-cap first becomes much better defined

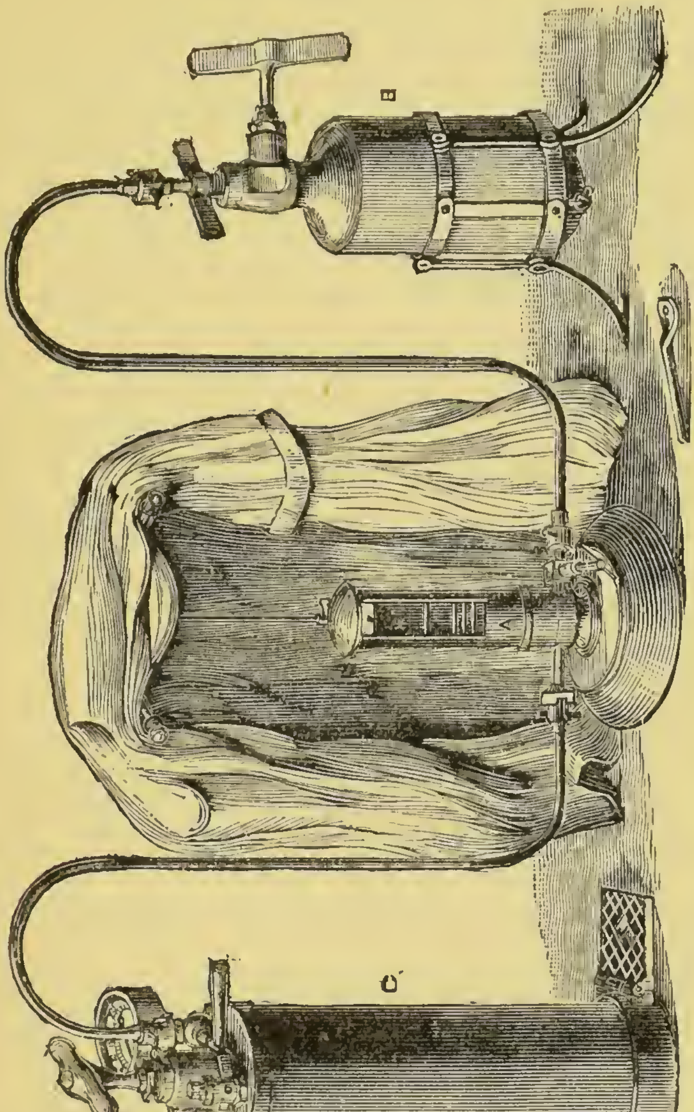


Fig. 27.—Apparatus for detecting Petroleum Vapour.

though it is not greatly augmented in size, and then considerable enlargement of the cap occurs, this condition being arrived at before the atmosphere becomes inflammable.

"In taking a sample of the air in a tank the collecting vessel may be used in the tank if the proportion of vapour present is known to be small; but even in such cases it is better to employ a short suction-tube, the open end of which can be placed at the lowest point in the tank, where most vapour would probably be found. If, on the other hand, the atmosphere of the tank is suspected to contain so much vapour that there would be danger of its producing insensibility when taken into the lungs, and especially if the compartment is entered through a small man-hole, it would obviously be most improper that any one should be sent into the tank, and in that case the sample should be taken by the use of a long suction-tube reaching to the bottom."

If a sample of atmosphere gives no cap at all with this apparatus, absolute security may be felt in giving permission for work with naked lights in such air. It is important that the operator should assure himself that the connections from the pump to the interior of the lamp are quite free; also that all the baffles, &c., be periodically taken out and dust removed, as the test is worse than useless unless a free current of the atmosphere passes through the lamp.

Capillary Test.*—A method of measuring the capillary power of kerosenes and of lamp-wicks has been designed by Mr. Robert Redwood.

A trough of considerable capacity is supported in a cupboard, which must be maintained at a uniform temperature, at a height which permits of the shorter ends of the bent wicks dipping into it and the longer

* Redwood, "Petroleum," p. 542.

ends hanging outside over small beakers. The oil in the trough is thus allowed to syphon over, and the relative rate of flow gives the measure required. The results are comparative only.

For **the testing of wicks** the procedure is as follows : Fill the trough to within a quarter of an inch of

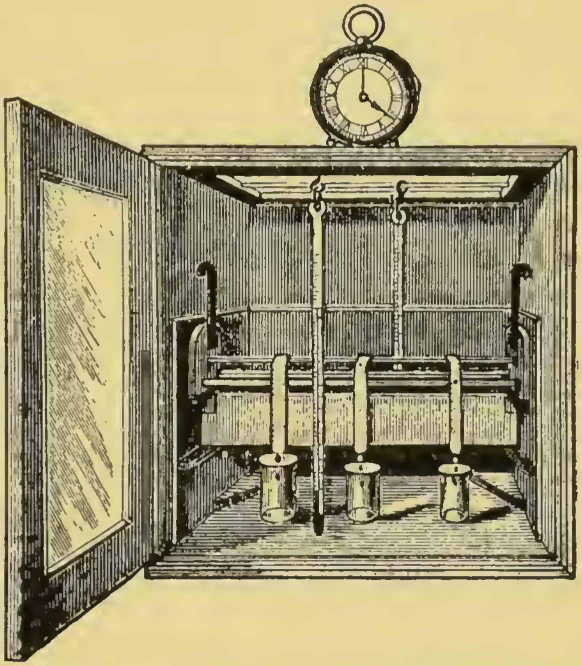


FIG. 28.—Redwood Apparatus for Capillary Test.

the top and bring the cupboard and oil to 70° F. Cut the wicks to be compared to exactly the same length (say 7 inches), and draw across them a light line in ink at the same distance from one end (say 3 inches). Then, having soaked them in the oil to be used and having removed all surplus oil by slight pressure, hang them on the highest of the three glass rods provided, and pass their ends *inside* the lower rods,* adjusting the ink marks so that they exactly correspond with the top of the upper rod.

Take as many small tared beakers as there are wicks

* The sketch is in error in this respect.

to be examined, and place them in front of the trough ready for use. Then put the wooden holders of the glass rods on the ledges at the end of the trough, allowing the short legs of the wick-syphons to dip into the oil. Note the exact time that the first drop falls from each wick, and remove each beaker at the end of thirty minutes from the time the first drop fell into it. The several weights of oil the beakers contain will then be a relative measure of the oil-passing capacity of each wick.

For testing oils, a trough divided into compartments must be used, and wicks of uniform character must be employed. In order to eliminate errors likely to be introduced by unavoidable differences in the wicks, the experiment may be repeated several times, using fresh wicks, and the average results given.

Melting-Point of Paraffin Scale and Wax.—The melting-point of the solid products of crude petroleum is taken commercially by two methods, known as the "English" and "American" tests.

In performing the first, the scale or wax is put into a test-tube 4 inches long by $\frac{3}{4}$ inch in diameter and melted in a water-bath. When complete liquefaction has taken place the tube is removed and its contents continually stirred with a delicate thermometer, the bulb of which is well covered by the sample. It is important that this stirring should be confined to the centre of the tube as much as possible, as contact with the walls of the tube may chill the bulb below the true temperature of the liquid. The fall in the mercury is arrested at about the same time that the liquid becomes cloudy, and an exact reading (to the nearest quarter of a degree F.) is taken at the first check in the cooling and recorded as the English Melting-Point.

The American test usually gives results $2\frac{1}{4}°$—$2\frac{3}{4}°$ F. higher than the English. The melted sample is poured into a warm metal dish hemispherical in shape and of $3\frac{3}{4}$ inches diameter. This is placed on a stand and a round-bulb thermometer (bulb $\frac{1}{2}$ inch

in diameter), supported in it with three-quarters of its bulb immersed, the whole being placed within a glass screen to avoid draughts. On carefully watching the sample, patches of film will be presently observed on its surface, and the temperature shown by the thermometer is noted at which these patches touch the bulb.

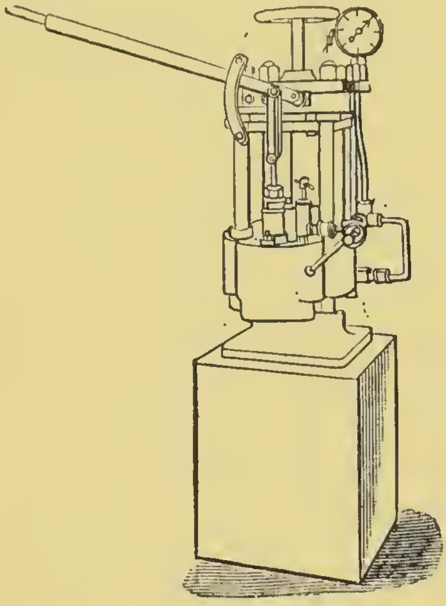


FIG. 29.—Hydraulic Press.

It will be observed that both the methods in general use give, really, the solidifying-point rather than the melting-point.

Oil in Scale.*—Five hundred grains of the powdered sample are weighed into a glass dish and transferred to a press. On each side of the scale is a circular calico press-cloth, and outside the cloths enough circular pieces of blotting-paper to absorb the oil expressed.

The diameter of the press-cup is $5\frac{5}{8}$ inches, and a total weight of 9 tons is applied for five minutes. The press may be of hydraulic power such as that figured.

* Redwood, "Petroleum," p. 631.

On the expiration of the time mentioned the press cake is again weighed together with the glass dish, and the carefully removed scrapings from the cloths. The loss in grains divided by five represents the oil percentage of the sample. The press and scale must be kept at 60° F. for the time of the experiment.

Estimation of Sulphur.—After numerous experiments carried out under the instructions of Sir Boverton Redwood with very varied methods it is clearly evident that while one method may be suited to one class of oil and another to another class, the Bomb-Calorimeter is the most trustworthy apparatus for employment with all oils. Full details as to the working of this instrument are given under the head of *Calorific Value* (p. 67).

After the ignition of the sample as there prescribed, the bomb is connected by a piece of rubber tubing to the bottom of a cylinder of glass beads moistened with weak alkali solution (free from sulphur), and the gaseous products of combustion are washed free from sulphates before escaping into the air. The bomb is opened, the washings poured into it, and then through a quick filter into a beaker together with three or four rinsings with distilled water. The usual precipitation with barium is then carried out.

Estimation of Water.—Samples that lose an inappreciable percentage on being heated to 230° F. may have their water contents estimated by weighing, say, 25 grammes into a glass dish, heating to that temperature on a sand-bath, and stirring continuously with a thermometer, until bubbles of steam cease to form. The sample is then allowed to cool, is reweighed, and the loss of weight in grammes multiplied by four represents the percentage of water.

In oils having more volatile constituents the water may be determined by subsidence in a Sutherland bulb (see Fig. 30). The sample is weighed into the bulb and the stopper tied in, and covered with a piece of rubber tissue to avoid the entrance of condensed steam.

The bulb is then placed in a bath kept at about 180° F. until no more water settles out. Oils of high viscosity may be diluted with kerosene to hasten the subsidence.

Or by distilling carefully over a naked flame from a tubulated retort, the water in, say, 250 c.c. may be collected in a c.c. measure and calculated to percentage.

Calorific Value.—The question of the thermal efficiency of oils is occupying the attention of the petroleum specialist to an increasing extent, in view of the steadily extending use of liquid fuel for industrial purposes. The apparatus yielding the most satisfactory results with a minimum of trouble is undoubtedly some form of "bomb calorimeter" such as that employed by M. Berthelot. The high cost of this particular instrument, owing to the amount of platinum used in its construction, often places it beyond the reach of the chemist, but a very good substitute has been designed by Mahler, and may be safely recommended.

FIG. 30.
Sutherland Bulb.

The following free translation from the French of the pamphlet supplied by the makers gives full guidance as to its manipulation.

The calorimeter is essentially composed of a bomb *B*, a calorimeter *D*, an insulating jacket *A*, and an agitator *S*. The bomb has a capacity of about 650 c.c., and the walls are 8 mm. thick. This capacity assures complete combustion of the sample by providing a decided excess of oxygen.

The bomb, made of specially forged steel, is nickel-plated outside, and has an inner coating of enamel*

* The specific heat of steel is 0·1150; this specific heat has been determined by M. Matignon, at the College of France (laboratory of M. Berthelot). The specific heat of enamel is 0·2045, also found by M. Matignon.

to resist the corrosive action of the nitric acid which always forms during the combustion. The bomb is closed by a screw lid fitting tightly on a lead washer. The cover carries a screw valve, which permits the introduction of oxygen.* Through the cover (or lid) a platinum electrode *E* (well insulated) is carried into the interior of the bomb.

Another rod of platinum is also fixed, supporting the capsule plate *E*, where the sample to be tested is placed.

The sample is ignited by contact with the iron wire spiral *F* (of known weight), which is connected at the desired moment to an electric current of about 12 volts and 2 amperes. A simple mechanical combination works the helicoidal agitator, and allows the operator to give a regular stirring movement without trouble. On the right side of the stand carrying the manometer is placed a screw valve, which can easily be adjusted to allow the slow introduction of the oxygen into the bomb. This valve avoids the necessity of using the valve on the oxygen cylinder, and is much more easily adjustable to give a slow supply of oxygen. M. Mahler uses the cylinders of compressed oxygen as figured, and as a suitable pressure for the combustion of 1 gramme of oil is 25 atmospheres, a cylinder containing 1000 litres of gas at 120 atmospheres would be sufficient for about 60 experiments.

Weigh one gramme of the substance to be tested into the capsule *C*, adjust the iron wire spiral used for igniting the sample. After the capsule has been firmly fixed on its supporting rod, screw on the lid of the bomb very firmly by gripping the latter in the vice provided and using the large spanner. Connect the screw valve of the bomb to the tube leading from the pressure gauge, taking care to have the screw valve on the manometer stand closed. First open the tap

* The small nut on the top of the milled head is provided to keep the screw valve in position by means of a spanner when the tube is screwed on.

of the oxygen cylinder *O*, and then very gently open the screw tap on the manometer stand, and allow the

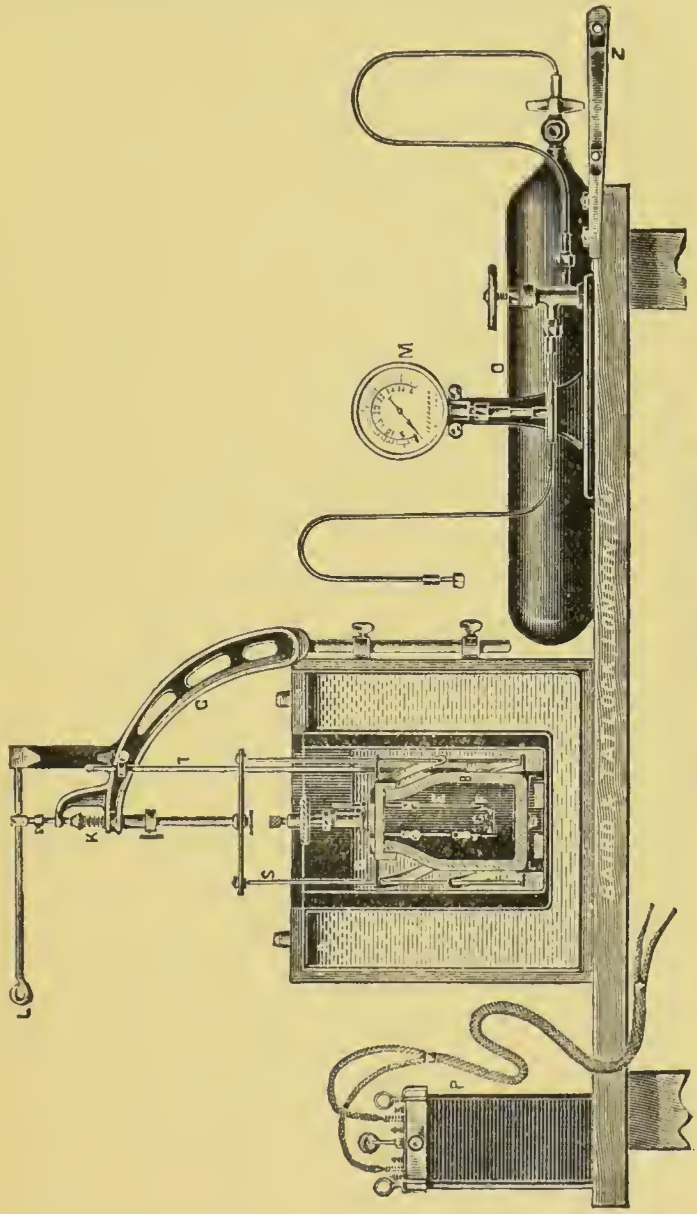


FIG. 31.—Mahler Calorimeter.

gas to flow until it registers a pressure of 25 atmospheres on the manometer. Now close the manometer screw tap, the screw tap of the oxygen cylinder, also the

screw tap on the cover of the bomb ; and disconnect the tube from the bomb. It is recommended that the substance to be tested should not be in too fine a powder, as the current of gas entering may blow it out of the capsule. To avoid this, care should be taken to allow the oxygen to enter the bomb slowly.

Place the bomb in the calorimeter A, then put the thermometer *T* and the agitator *S* in position, and pour in the water which has been previously measured. Leave the apparatus so assembled for a few minutes, stirring at intervals so as to ensure a somewhat equable temperature before commencing observations. Then, when all is ready, the stirring is performed continuously, and a careful note taken of the temperature every minute for about five minutes, so as to fix the law which the thermometer follows before the substance is ignited. Now ignite the substance by connecting one wire of the battery to the platinum electrode *E*, and the other wire to some part of the screw tap. Take note of the temperature half a minute after the ignition, also at the end of this minute and continue to take the thermometer readings every minute until they commence to show a regular fall in temperature. This is the maximum temperature to be noted. Continue the readings of the thermometer for another five minutes, so as to fix the law the thermometer follows after the maximum temperature has been reached.

The following are the rules for correction of calculation : (1) The law of the decrease of temperature observed, after the maximum, represents the loss of heat of the calorimeter before the maximum for any one minute, on the condition that the average temperature of this minute does not differ by more than one degree from the maximum temperature. (2) If the temperature of the examined period differs by more than one degree, but by less than two degrees from that of the maximum, the figures which represent the law of decrease at the moment of the maximum, diminished by 0·005, give the correction required. The

two preceding remarks suffice in all cases. Moreover, without affecting the precision of the experiment, it may be taken that the law of variation followed during the first half of the minute during which the ignition took place is that which existed at the beginning of that minute. During the whole time of the observation the operator ought to take care to see that the agitator acts regularly.

When the observation is finished, first open the screw tap, then the bomb itself. Now wash the interior of the bomb with a little water, so as to collect the acid formed during the explosion. The nitric acid is estimated by titration with alkali, and now we are in possession of all the elements for the calculation, for since the calorific power Q is the whole,

$$Q = \Delta (P + P') - (0{\cdot}23\, p + 1{\cdot}6\, p')$$

Δ = the difference of the corrected temperature.
P = the weight of water in the calorimeter.
P' = the equivalent in water of the bomb and its accessories.
p = the weight of the nitric acid (HNO_3) found.
p' = the weight of the small iron spiral.
0·23 = the heat caused by the formation of one gramme diluted nitric acid.
1·6 = the heat caused by the combustion of one gramme of iron.

If it is a question of testing oil, no notice need be taken of the small quantity of sulphuric acid which results from the oxidation of the sulphur of the sample, and which will be estimated in the titration as nitric acid. The error is in reality negligible in a commercial estimation. But one will notice that the bomb gives a means of estimating the sulphur, which is entirely oxidised and transformed into sulphuric acid.* In this case it is unnecessary to take note of the thermometer reading. In the case of a test of a substance with little hydrogen in it, coke for example, so little water is formed that the quantity is insufficient to dissolve the acids. It is then necessary to put at the

* The heat given out by the formation of the diluted sulphuric acid H_2SO_4 can be calculated on the basis of 0cal73 per gramme of H_2SO_4.

bottom of the bomb a few c.c. of water, which one must take into account in the calculation.

Proceed just the same for a liquid as for a solid. But if the liquid emits appreciable vapours at ordinary temperatures it is better to weigh the amount taken in a thin capsule with slender points, through which passes the fuse of iron wire. When the capsule is introduced into the bomb, care must be taken to break these points, so as to allow the oxygen to make contact with the liquid.

The method of finding the calorific power of gases is as follows: Carefully measure the empty space in the bomb, fill it first of all with gas, now empty, and definitely introduce the gas under atmospheric pressure at the temperature of the laboratory, then add the oxygen, and proceed the same as for solids or liquids.

The determination of the calorific power of gases offers a peculiar difficulty. Care must be taken not to mix the gas with such a quantity of oxygen that the mixture ceases to be explosive. For ordinary lighting gas, 5 atmospheres of oxygen are sufficient. For the gas of Siemens' generator not more than half an atmosphere, measured by the mercury manometer, must be taken.

Determination of the Equivalent in Water of the System.—To find the term of correction representing the exact equivalent P' of the system in water the most simple way is to make a double experiment as follows: Burn in the bomb a known weight, one gramme, of naphthalene for example, with 2300 grammes of water in the calorimeter. Next burn one gramme of naphthalene with only 2100 grammes of water in the calorimeter. We have, then, two equations which between them eliminate the calorific value of the naphthalene, and the water equivalent can be deduced from the difference in the two sets of figures. Care must be taken to weigh the naphthalene after it is half melted, otherwise, if it were not fused, some of it would be scattered by the current of oxygen, and would not be burnt.

Example of the Determination of Calorific Value.—The combustible was a sample of colza oil; its analysis gave: Carbon, 77·182%; hydrogen, 11·711%; oxygen and nitrogen, 11·107%. The weight taken was one gramme. The calorimeter contained 2200 grammes of water. The equivalent in water of the bomb and its accessories was 481 grammes.*

The apparatus having been assembled as described, a few minutes were allowed to elapse, so as to establish a uniform temperature, the stop-watch was started, and the thermometer readings recorded as below:

Preliminary Period.

0 minutes	10·23°
1 ,,	10·23°
2 ,,	10·24°
3 ,,	10·24°
4 ,,	10·25°
5 ,,	10·25°

$$\Delta_0 = \frac{10{\cdot}25^\circ - 10{\cdot}23^\circ}{5} = 0{\cdot}004$$

ignited by means of electrodes.

Period of Combustion.

5½ minutes	10·80°
6 ,,	12·90°
7 ,,	13·79°
8 ,,	13·84° (maximum)

Last Period.

9 minutes	13·82°
10 ,,	13·81°
11 ,,	13·80°
12 ,,	13·79°
13 ,,	13·78°

$$\Delta T = \frac{13{\cdot}84^\circ - 13{\cdot}78^\circ}{5} = 0{\cdot}012$$

* The equivalent in water, 481 grammes, was found by a special method giving directly the exact heat value of the system.

The thermometrical observations were stopped. The variation of the temperature was:

$$13{\cdot}84^\circ - 10{\cdot}25^\circ = 3{\cdot}59^\circ$$

Take note of the following corrections. The system had lost, during the minutes (7, 8) (6, 7), a quantity of heat =

$$\frac{13{\cdot}84^\circ - 13{\cdot}78^\circ}{5} \times 2 = 0{\cdot}012 \times 2 = 0{\cdot}024 \; *$$

During the half-minute ($5\frac{1}{2}$, 6) a quantity of heat =

$$(0{\cdot}012 - 0{\cdot}005)\,\tfrac{1}{2} = 0{\cdot}0035$$

and during the half-minute (5, $5\frac{1}{2}$) it gained

$$\frac{10{\cdot}25^\circ - 10{\cdot}23^\circ}{5} \times \tfrac{1}{2} = 0{\cdot}004 \times \tfrac{1}{2} = 0{\cdot}0020 \; \dagger$$

It follows that the net loss during the minute (5, 6) was $0{\cdot}0035 - 0{\cdot}002 = 0{\cdot}0015$.

Altogether the system lost during the experiment a quantity of heat $= 0{\cdot}024 + 0{\cdot}0015 = 0{\cdot}0255$, which must be added to the $3{\cdot}59^\circ$ already found.

The variation of the corrected temperature is then $3{\cdot}615^\circ$, neglecting the fourth place. The quantity of heat observed is therefore $(2{\cdot}200 + 481) \times 3{\cdot}615 = 9^{cal}\,691815$. Take $9^{cal}\,6918$.

To get the required result we must subtract from this:

(1) The heat caused by the formation of 0·13 gr. of nitric acid HNO_3, titrated volumetrically .	0·13	× 0·23	= 0 cal 0299
(2) The heat of the combustion of 0·025 gr. of iron thread . .	0·025	× 1·6	= 0 cal 0400
To be subtracted . .			0 cal 0699

* Law of cooling after the maximum.

† Law of variation of temperature at the moment of the minimum.

The final result is then :

$$9^{cal}\ 6918 - 0^{cal}\ 0699 = 9^{cal}\ 6219$$

or for one kilogramme of oil $9{\cdot}621^{cal}\ 9$.*

* Note that the dimensions of the apparatus are such that one can, without difficulty, arrange once for all so that in all experiments the insignificant corrections cancel one another. If x is the correction due to loss of heat during the operation, it will not be necessary to make the correction if

$$0{\cdot}23p + 1{\cdot}6p' = x(P + P').$$

p' is at the disposition of the worker within certain limits ; P equally so. Evidently, then, it is possible to make the equation sufficiently true. Thus the calorific value of colza oil is $9621{\cdot}9^{cal}$. Simply multiplying 3·59 by 2681 gives 9624, which approximates to within 2 in 9000.

REDWOOD VISCOMETER, ADMIRALTY PATTERN

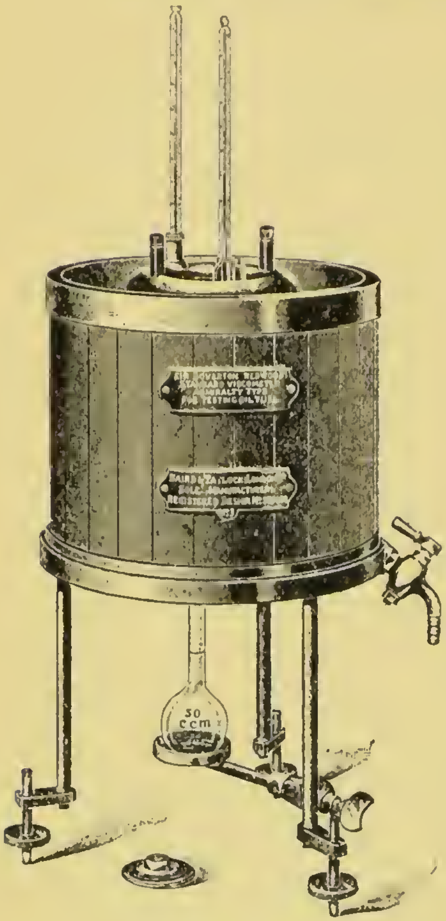


FIG. 32.

A modified form of the Redwood Viscometer, for use in connection with the Admiralty specification for oil fuel, has recently been devised by Sir Boverton Redwood. The construction of the instrument is shown in the accompanying illustration. The design is registered, and the sole makers are Messrs. Baird & Tatlock (London), Ltd. The directions for use are as follow:

The oil to be tested must be free from water, dirt, and matter in suspension, and should be maintained at a temperature of 32° F. for at least six hours immediately before it is tested.

The thermometer is supported in the oil-cup by means of the clip, so that its bulb rests on the bottom of the cup, and in such a position that the flow of oil from the orifice will not be interfered with.

The oil-cup is filled with the cooled oil, so that the gauge-point is covered, and the apparatus is then transferred to an efficient ice-chest of suitable design and the instrument levelled by means of the tripod-screws and spirit-level provided for that purpose.

As it may be necessary to make more than one test, it will be found desirable to have at least half a pint of the oil in a stoppered glass bottle placed in the ice-chest with the instrument until required for use.

Before commencing a test, the level of the oil in the oil-cup is brought exactly to the point of the gauge. A narrow-necked flask, of 50 cubic centimetres capacity to a mark on the neck, is placed beneath the jet and the ball valve raised and suspended from the thermometer-clip by means of the hook on the stem of the valve, a stop-watch being started simultaneously with the lifting of the valve. During the period of testing, the temperature of the oil must remain constant at 32° F. The time of outflow for 50 c.c. is noted, and the result given in seconds.

If the oil-cup requires to be wiped out, tissue-paper rather than cloth should be employed, as filaments of the latter may be left adhering to the surface of the vessel. The agate orifice must not be cleaned with anything which is likely to cause abrasion, and it has been found best to employ twisted tissue-paper for the purpose.

APPENDIX

TABLE FOR THE CONVERSION OF CENTIGRADE DEGREES INTO FAHRENHEIT DEGREES.

° C.	0	1	2	3	4	5	6	7	8	9
0	32	34	36	37	39	41	43	45	46	48
10	50	52	54	55	57	59	61	63	64	66
20	68	70	72	73	75	77	79	81	82	84
30	86	88	90	91	93	95	97	99	100	102
40	104	106	108	109	111	113	115	117	118	120
50	122	124	126	127	129	131	133	135	136	138
60	140	142	144	145	147	149	151	153	154	156
70	158	160	162	163	165	167	169	171	172	174
80	176	178	180	181	183	185	187	189	190	192
90	194	196	198	199	201	203	205	207	208	210
100	212	214	216	217	219	221	223	225	226	228
110	230	232	234	235	237	239	241	243	244	246
120	248	250	252	253	255	257	259	261	262	264
130	266	268	270	271	273	275	277	279	280	282
140	284	286	288	289	291	293	295	297	298	300
150	302	304	306	307	309	311	313	315	316	318
160	320	322	324	325	327	329	331	333	334	336
170	338	340	342	343	345	347	349	351	352	354
180	356	358	360	361	363	365	367	369	370	372
190	374	376	378	379	381	383	385	387	388	390
200	392	394	396	397	399	401	403	405	406	408
210	410	412	414	415	417	419	421	423	424	426
220	428	430	432	433	435	437	439	441	442	444
230	446	448	450	451	453	455	457	459	460	462
240	464	466	468	469	471	473	475	477	478	480

TABLE FOR THE CONVERSION OF CENTIGRADE DEGREES INTO FAHRENHEIT DEGREES—(*continued*)

° C.	0	1	2	3	4	5	6	7	8	9
250	482	484	486	487	489	491	493	495	496	498
260	500	502	504	505	507	509	511	513	514	516
270	518	520	522	523	525	527	529	531	532	534
280	536	538	540	541	543	545	547	549	550	552
290	554	556	558	559	561	563	565	567	568	570
300	572	574	576	577	579	581	583	585	586	588
310	590	592	594	595	597	599	601	603	604	606
320	608	610	612	613	615	617	619	621	622	624
330	626	628	630	631	633	635	637	639	640	642
340	644	646	648	649	651	653	655	657	658	660
350	662	664	666	667	669	671	673	675	676	678
360	680	682	684	685	687	689	691	693	694	696
370	698	700	702	703	705	707	709	711	712	714
380	716	718	720	721	723	725	727	729	730	732
390	734	736	738	739	741	743	745	747	748	750
400	752	754	756	757	759	761	763	765	766	768
410	770	772	774	775	777	779	781	783	784	786
420	788	790	792	793	795	797	799	801	802	804
430	806	808	810	811	813	815	817	819	820	822
440	824	826	828	829	831	833	835	837	838	840
450	842	844	846	847	849	851	853	855	856	858
460	860	862	864	865	867	869	871	873	874	876
470	878	880	882	883	885	887	889	891	892	894
480	896	898	900	901	903	905	907	909	910	912
490	914	916	918	919	921	923	925	927	928	930
500	932	934	936	937	939	941	943	945	946	948

INDEX

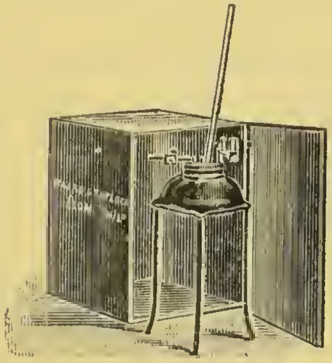
BALLANTYNE & COMPANY LTD
TAVISTOCK STREET COVENT GARDEN
LONDON

BAIRD & TATLOCK (LONDON), LTD.

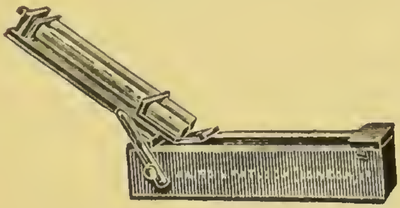
LIST OF APPARATUS REQUIRED FOR OIL TESTING, &c., AS MENTIONED IN THE "LABORATORY BOOK OF MINERAL OIL TESTING," BY JAS. A. HICKS.

Special Catalogue on Application.

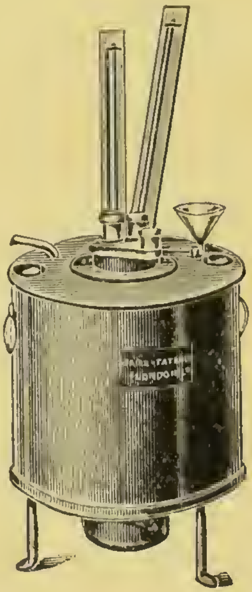
All apparatus detailed below is required for a full equipment ; the quantity will vary according to requirements.



No. 19



No. 32



No. 11

SPECIFIC GRAVITY.

Item		Page
1.	Hydrometer, specific gravity	4
2.	Hydrometer, thermometer for	4
3.	Hydrometer Jar	4
4.	Specific Gravity Bottle with drilled stopper	6
5.	Thermometer for Specific Gravity Bottle	6
6.	Regnault's Specific Gravity Bottle	8
7.	Sprengel Tube	9
8.	Water-Bath for Sprengel Tube	9
9.	Westphal's Specific Gravity Balance	10
10.	Sartorius' Specific Gravity Balance	11

FLASHING-POINT.

11.	Abel's Flash-Point Apparatus	13
11a.	Thermometers for ditto	13
11b.	Abel's Flash-Point Apparatus with Agitator (Fig. 8)	19